PATENT ABSTRACTS OF JAPAN

(11)Publication number:

11-343152

(43)Date of publication of application: 14.12.1999

(51)Int.Cl.

CO3C 27/12

(21)Application number: 10-203425

(71)Applicant: SEKISUI CHEM CO LTD

(22)Date of filing:

17.07.1998

(72)Inventor: SHICHIRI NORISHIGE

MIYAI JIRO

BANDO AKIHIKO TOYAMA KIYOBUMI SANNOMIYA YOSHINARI NAKAJIMA MINORU

AOSHIMA YOSHIO

(30)Priority

Priority number: 09192474 Priority date: 17.07.1997 Priority country: JP 09213284 07.08.1997 JP 09223613 20.08,1997 JP 09247015 11.09.1997 Jp 09253216 18.09.1997 Jp 10 983 06.01.1998 JP 10 22137 03.02.1998 JP 10 91947 03.04.1998 JP

(54) INTERMEDIATE FILM FOR LAMINATED GLASS AND LAMINATED GLASS

(57) Abstract:

PROBLEM TO BE SOLVED: To obtain a laminated glass which maintains basic performance necessary for a laminated glass and little efflorescence in the peripheral part even in a high humidity atmosphere by forming an intermediate film comprising a plasticized polyvinyl acetal resin film having a specified or lower haze when the film is immersed in water.

SOLUTION: When the intermediate film for a laminated glass comprising a plasticized polyvinylacetal resin film having 0.3 to 0.8 mm thickness is immersed in water at 23° C, efflorescence of the film after 24 hours is controlled to ≤50 % haze. To obtain the haze value above described, sodium salts and potassium salts which cause the efflorescence and which are mixed from the source material are controlled to have ≤10 µm particle size and to give ≤50 ppm sodium conon, and ≤100 ppm potassium concn. in the intermediate film. To effectively prevent efflorescence at high humidity, compds, which can form complexes with sodium salts and potassium salts, and org, acids and amines compatible with the resin and the plasticizer are preferably added as dispersants.

11-343152A [OLAIMS]

ペープス

* SECTION

and IMPIS are not responsible for any

his document has been translated by computer. So the translation may not reflect the original ages caused by the use of this translation.

*** shows the word which can not be translated. cisely

the drawings, any words are not translated.

sim(s)]

aim 1]An interlayer for glaas laminates when it is an interlayer for glass laminates which consists slasticized polyvinyl polyvinylacetal resin films and said 0.3–0.8-mm-thick interlayer is immersed in ** water, wherein Hayes of 24 hours after is 50% or less.

sim 2]The interlayer for glass laminates according to claim 1 whose particle diameter of sodium

aim 3]The interlayer for glass laminates eccording to claim 1 or 2 whose particle diameter of in an interlayer is 10 micrometers or less.

aim 4) The interlayer for glass laminates according to claim 1, 2, or 3 whose sodium concentration fum salt in an interlayer is 5 micrometers or less. in interlayer is 50 ppm or less.

sim 5]The interlayer for glass laminates according to claim 1 whose particle diameter of potassium in an interlayer is 10 micrometers or less.

aim 6]The interlayer for glass laminates according to claim 1 or 5 whose particle diameter of

assium salt in an interlayer is 5 micrometers or less.

aim 7]The interlayer for glass laminates according to claim 1, 5, or 8 whose potassium

aim 8] The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 8, or 7 which is a thing training a compound which can form sodium salt and potassium salt, and a complex. scentration in an interlayer is 100 ppm or less.

aim 9]The interlayer for glass laminates according to claim 1. 2, 3, 4, 5, 6, or 7 which is a thing

eim 10]The interlayer for glass laminates according to cleim 1, 2, 3, 4, 5, 6, 7, 8, or 9 which is a Ig containing at least one sort chosen from a group which consists of akali metal saft and alkaline taining amine which is compatible in organic acid which is compatible in resin and a plasticizer; in, and a plasticizer.

h a particle diameter of 3 micrometers or less and whose alkaline earth metal salt is a thing with a aim 11] The interlayer for glass laminates according to claim 10 whose alkali metal salt is a thing ticle diameter of 3 micrometers or less. th metal salt

all metal saft of organic acid of the carbon numbers 5-16 and whose alkaline earth metal saft is the aim 12)The interlayer for glass laminates according to claim 10 or 11 whose alkali metal salt is the aline earth metal saft of organic acid of the carbon numbers 5-15.

aim 133A glass laminate characterized by making the interlayer for glass laminates according to m 1, 2, 3, 4, 5, 8, 7, 8, 8, 10, 11, or 12 come to intervene between glass of a couple at least.

anstation done.]

o.//www4.ipdl.inpit.go.jp/cgi-bin/tran_web_cgi.ejje?atw_u=http://www4.ipdl.inpit.go.jp.__2009/06/15

* SECTION

ages caused by the use of this translation. and iMPIT are not responsible for any

his document has been translated by computer. So the translation may not reflect the original cisely.

*** shows the word which can not be translated.

the drewings, any words are not translated.

TAILED DESCRIPTION

tailed Description of the Invention]

sid of the Invention.] This invention relates to the giass laminate which used the interlayer for glass inates, and the above-mentioned interleyer for glass laminates. 03

as so that it may fall within a proper range. Namely, if the danger of the glass fragment damaged by nan body etc. if the adhesive strength of an interlayer and glass is too weak becomes high and the ulteneously damaged by the shock from the outside, etc., and the danger of the echesion fragment wide and happans, the impact-absorbing effect and a penetration preventive effect become large. ic performance is good and excellent in safety. That is, by the pariphery of a glass laminate, when rlayer which consists of a plasticization polywind butyral between the glass plates of at least two above-mentioned glass faminate is placed into a humid atmosphere, since the interlayer touches an interlayer extends arises at the same time glass is damaged while glass breakage reaches far cribed below is participating in this albinism. In order to demonstrate the function as the abuve ntioned glass laminate enough, it is required to adjust the adhesive strength of an interlayer and ts. Since the phenomenum in which partial interfacial peeling of an interlayer and glass happens. shook from the outside, etc. separating and dispersing from an interlayer, and doing injury to a ets. It has basic performance transparency, weatherability, and an adhesive property are good, 03] This kind of glass laminate is inferior in maisture resistance, although the above-mentioned 05)On the other hand, when there is adhesive strength of an interlayer and gass within proper id penetration resistance moreover and required for the glass faminate of a glass fragment not scription of the Prior ArtiThe glass laminate with which it comes conventionally to fasten the lesive strength of an interlayer and glass is too strong conversely, Glass and an interlayer are 04]The additive agent for performing adhesive strength adjustment of an interlayer and glass dass and an interlayer dispersing and doing an obstacle to a human body etc. becomes high. persing easily, for example, is widely used for the windowpane of a cer or a building. ironmental air directly, the problem which the interlayer of a periphery milks arises.

orb a shock in case a driver end a passenger collide to glass or to prevent penetration, in case of mined variously. In JP,46-4270,B, the interlayer for glass laminates which consists of a polyvinylisture and the metal alkyl carboxylate specific as an adhesive strength regulator is proposed. The accident of a building, in order to prevent the missile from the outside from penetrating glass or 06] Therefore, in case of the accident of transport-sirplane machines, such as a car, in order to glass to the proper range by changing the distribution quantity of metal alkyl carboxylate in an ital resin composition which carries out specific amount content of 0.2 to 0.8 % of the weight of wayer by the above-mentioned proposal tends to adjust the adhesive strength of an interlayer stayer layer part and an interfayer inner layer part, or changing the moisture content in en prevent scattering of a glass fragment, it is required to adjust the adhesive strength of an 07 lin order to adjust the adhesive strength of an interlayer and glass within proper limits wentionally in view of the above, the adhesive strength regulator for interlayers has been arisyer and glass so that it may fall within a range proper like ****

08]Howaver, the interlayer containing metal alkyl carboxylate like the above-mentioned proposal,

21/wwwf.ipdl.inpit.go.jp/cgi-bin/tran.web_cgi.ejie?atw_u-http%3A%2F%2Fwww4.ipdil... 2009/06/15

JP.11-343152.A [DETAILED DESCRIPTION]

under a humid atmosphere, While the quantity of metal alkyf carboxylate increases, there is a problem glass laminate which moisture resistance fell and was manufactured using this interlayer is neglected carboxylate as much as possible, and by losing, in that case, the fatal problem as a glass laminate of Since the interlayer is carrying out direct contact to air in the periphery of the glass laminate if the the achesive strength of an interlayer and glass becoming stronger than the proper range too much, that the albinism by moisture absorption of an interlayer happens violently. Although it can prevent and glass and an interlayer being simultaneously damaged by the shook from the outside, etc., or whether the albinism of the above-mentioned interlayer reduces the quantity of metal alkyl becoming easy to penetrate had occurred.

0.01~3 weight~section content of the aliphatic series monoamine dicarboxylic acid of four to 5 narbon (0009]In JP,44-32185,8, contain 0.1 to 0.8% of maisture, and The monocarboxyliz soid of six to 22 monocarboxylic acid of two to 6 carbon atom. The interlayer for glass laminates which carries out carbon atom. The dicarboxylic acid of four to 12 carbon atom, and the alighatic series monoamino atom and at least one organic acid chosen from olirate and these mixtures per resin 100 weight section and which consists of shaping polyvinyl-acetal rasin is propased.

with time. The problem that the heat resistance and the weatherability of an interlayer fall under the [0010]However, when carboxylic acid is added, there is a problem that adhesive strength changes influence of acid arises.

polyvinyl-acetal-resin censtituent in JP,48-5772,B. The glass laminate making sodium metal sait of the alighatic carboxylic acid of the carbon numbers 10-22 contain in this plasticized-polyviny!-(0011) In the glass which pasted the glass of at least two sheets together with the plasticizedacetal-resin constituent is proposed.

or dicarboxylic acid is proposed as an achesive strangth regulator in the plasticized-polyvinyl-acetal-[0012]in 3P,53-18207,B, using the alkali metal salt or alkaline earth metal salt of monocarboxylic acid resin interlayer of a glass laminate.

comparatively large carbon number as an adhesive strength regulator from it being easy to dissolve in 10013 In the two above-mentioned prepasals, each uses metal sait of cerboxylic acid with a the plasticizer contained in an interlayer.

(DO14)However, when metal salt of carboxylic acid with a large carbon number is used as an adhesive declines gradually with temporality, and when shocked, glass extoliates easily. In order to prevent this with time progress (temporality). That is, even if early adhesive strength is proper, adhesive strength keeping it under the above atmosphere for a long period of time rines difficult actually, although the strength regulator, there is a problem that the adhesive strength of an interlayer and glass changes atmosphere, and to ripe, and Since an interlayer has adhesiveness, autohesion nature, etc., even if fall of adhesive strength with the passage of time can be controlled, it cannot be made for there to adhesive strangth fall, it is necessary to keep an interlayer for one to two months under 40-50 ** be nothing, but the above-mentioned problem will still remain.

(0015]In JP,60-210551,A, to plasticized-polyvinyl-acetel-resin 100 weight section. The glass laminate used and this glass laminate caused a white blush mark, the perfect thing was not able to say from a whose carbon numbers are 1-6 contain, and the interlayer to which it, adheres is indicated. However, potassium monocarboxylate 0.40 weight section and 0.01 to modified silicone oil 0.26 weight section since this metal salt solidified in particle state in the interlayer depending on the kind of metal salt with which the glass of at least two sheets sticks, and it comes to unite it by whether 0.02 to viewpoint of preventing the white blush mark by long-term moisture absorption.

10016]in JP,2-41547,8, the polyvinyl-butyral sheet which uses alkali or alkelina-earth-metals formate for an achesive strength regulator is proposed. In the Patent Publication Heisei No. 502594 [six to] gazeite, the interlayer which added potassium acetate as an adhesive strength regulator in the example is used.

metal sait of carboxylic acid with a large carbon number as an adhesive strength regulator, metal sait 10017In the three above-mentlened proposals, in order to cancel said problem in the case of using of carboxylic acid with a comparatively small carbon number is used

interlayer and glass is canceled, the moisture resistance of an interlayer becomes insufficient, and, as strength regulator, Although the problem of an adhesive strangth fall with the passage of time of an a result, arruther problem of becoming easy to start the albinism by moisture absorption to the edge (0018) However, if metal sait of carboxylic acid with a small carbon number is used as an adhesive

http://www4.jodl.inpit.go.jp/ogi-bin/tran.web.ogi.ejie?etw.u=http%3A%2F%2Fwww4.jpdl.i..._2009/06/15

t (end) of a glass laminate occurs.

he weight. At this time, water gathers for the circumference of metal selt of carboxylic acid with a a small carbon number or its salt is decreased in order to reduce albaism, the adhesive strength example so that water content may be about 0.5 or less % of the weight under the atmosphere of sidity etmosphere, an interlayer absorbs moisture and water content usually rises to about 2 to 3% 18]That is, since hygroscopicity is under the usual atmosphere (rumidity), when processing it into isss laminate, it is common (an interlayer) to process it by controlling the humidity and doubling, its as a minute crystal in an interlayer, and albinism is caused. If the addition of carboxylic sold in interlayer and glass will deviate from the proper range, and will become insufficient { impactall carbon number of potassium acetate, magnesium acetate or potassium formate etc. which RH. Hawever, since the edge part of a glass laminate is in an unreserved state, under a high orption nature, penetration resistance, etc. of a glass laminate].

in JP,5-188250.A. The interlayer for glass laminates ourrently formed from polyvinyf-scetal resin, plasticizer, and the resin composition in which alighatic series mone- ** whose carbon number is 20]As a trial to improve, the white blush mark of the interlayer containing carboxylic acid metal or less contains the alkell or alkaline earth metal salt, and organic acid of dicarboxylic acid is posed.

taining polyvinyt-acetal resin, a plasticizer, carboxylic acid metal salt, and straight-chain fatty acid 21]The interlayer for glass faminates which becomes JP,7–41340,A from the resin composition reposed.

istance test is reduced. And when it is going to reduce a white blush mark further and the content 22]However, the glass laminate using the interlayer for glass laminates of the above-mentioned waight—chain fatty acid is increased, and a glass laminete is placed under relatively high posal is not still enough, although the white bash mark of the edge part after a humidity sperature, there is a possibility of producing feaming and discoloration.

esive strength regulator, the white blush mark by maisture absorption produces it. It became clear our latest research that the impurity in the resin described below is involving as one of the cause 23]Although the interlayer of the above-mentioned proposal tries solution of a white blush mark improvement of an adhesive strength regulator, also in the interlayer which is not adding the

per. Sodium saft may remain also to polyvinyl alcohol and this sodium saft may cause a white blush 24]Aithough the interlayer for glass laminates of this invention uses polyviny!-acetal resin as the wlayer for glass laminates obtained in order to promote condensation of water, when it becomes oe the solution of sodium salt, such as sodium hydroxide and sodium bicarbonate, is used in this n ingredients, the neutralization process is included when manufacturing polyvinyl-acetal resin. tralization process, for example, When those sodium salt is used superfluously and new sodium ticle state at the time of a polymerization and desiccation and polyvinyl-acetal resin absorbs s sodium saft that remained causes [hig] a white blush mark by moisture absorption of the generates by neutralization, sodium saft remains in the polyvinyl-acetal resin obtained. k by moisture absorption of the interlayer for glass laminates.

dings of a car prospers, in these uses, also when using the periphery of a glass laminate in the eserved state, it is increasing, and the request to elbinism prevention is increasingly stronger. 25]In recent years, the motion which uses a glass laminate for side glasses and the various

is faminate edge part in providing the interlayer for [fittle] glass laminates, and the glass laminate. istance, even when mareaver placed into a humid atmosphere, there is a white blush mark of a ationed problem and is made into the purpose, Without spoiling basic performance required for is laminates, such as transparency, weatherability, an adhesive property, and penetration oblam(s) to be Salved by the Invention] The place which this invention solves the above-

ians for Solving the Problem!This invention is an interlayer for glass laminates which consists of sticized polyvingl polyvinglacetal resin films, and when the 0.3-0.8-mm-thick above-mentioned stayer is immersed in 23 ** water, Hayes of 24 hours after is an interlayer for glass laminates ch is 50% or less. This invention is explained in full detail below. 2.//www4.jpdl.impit.go.jp/cgi-bin/tran_web_caj.ejje?atw_urhittp%3A%2F%2Fwww4.jpdli... 2009/06/15

[0028]When an interlayer for glass laminates of this invention immerses a 0.3-0.8-mm-thick interlayer

in 23 ** water, Hayes of 24 hours after is 50% or less.

[8029]When this invention persons inmerse a 0.3–0.8–mm-thick interlayer in 23 ** water, Even when leminates is humid, there were few white blush marks of a glass laminate edge part, they found out Hayes of 24 hours after was placed into atmosphere where 50% or less of interlayer for glass excelling in moisture resistance, and completed this invention.

10030]Since it becomes insufficient to prevent a white blush mark under high humidity and it is mentioned range. Above-mentioned Hayes as used herein shall mean a value which used and measured an intagraf equation turbidity metar 24 hours efterward, when a 0.3–0.8–mm—thiok inferior to moisture resistance when above-mentioned Hayes exceeds 50%, it is limited to a interleyer is immersed in 23 ** water.

18831]An interlayer for glass laminates of this invention consists of a plasticity polyvinyl-acetal resin layer, and the above-mentioned plasticity polyvinyl-acetal resin layer uses polyvinyl-acetal resin as

(0032) As the above-mentioned polyvinyl-anetal resin, average degree-of-acetalizations of a thing of mixing a plasticizer of a complement to reservation of penetration resistance --- hard --- ** --- there obtained will fall, in order to obtain resin, profonged reaction time is required, and it is not sometimes is a case. If 75-mol % is exceeded, while a mechanical strength of an interlayer for glass laminates manufacture of resin if 30-mol k is exceeded, it becomes difficult to manufacture. Preferably, it is [0033]]In the sbove-mentioned plasticized polyviny! acets! resin, the following [30 ms! %] have a 40-75 moi is preferred, compatibility with a plesticizer falling that it is less than [40 mol %], and hygrosoopioity becomes high, a white blush mark will happen easily. It is 64-71-mof % preferably. preferred vinyl acetate component. Since it will become easy to cause blocking at the time of desirable on a process. It is 80-75-mol % more preferably. If it is less than [60 mol %], since less than 19 mol %.

viryl stockol ingredient, and a vinyl acetate component, and each of these component amounts, it can measure based on for example, a JIS K 6728" polyvinyl-butyral test method" and a nuclear magnetic [0034]The above-mentioned plasticized polyvinyl acetal resin comprises a vinyl-acetal ingredient, a resonance method (NMR).

remaining vinyi-acetal component amounts can be computed by deducting buth the above-mentioned alcohol component amount and the amount of viry's acetate components can be measured, and the (0035)When the above-mentioned polyvinyl-acetal resin is except polyvinyl butyral resin, a vinyl component amounts from 100.

Subsequently, reaction temperature is raised to 70 **, it ripes, a reaction is completed, and a method [0036] The above-mentioned polyvinyl-apetal resin can be conventionally manufactured by a publicly of performing neutralization, rinsing, and desiccation and obtaining pewder of polyvinyl-acetal resin, acetalization reactions are advanced, adding and stirring a necessary acid catalyst and aldehyde. known method, for example, solution obtained by dissolving polyvinyl alcohol in warm water --- a predetermined temperature --- for example, it holds at 10-20 ** preferably, and 0-95 ** of etc. are mentioned after that.

[8837]As polyvinyl alcohol used as the above-mentioned raw material, a thing of the average degrees of polymerization 500-5000 is preferred, and a thing of the average degrees of polymerization 1000obtained may fall, if 5000 is exceeded, shaping of a resin layer will carry out --- hard --- ** --- there 2500 is more preferred. When it is less than 500, the penetration resistance of a glass laminate are things and, moreover, intensity of a resin layer may become strong too much.

0038]Since it is preferred to set a vinyl acetate component of polyvinyl-scatal resin obtained to leas As the above-mentioned aldehyde, aldehyde of the carbon numbers 3–10 is preferred. The moldability preferred thing beyond 70 mai %. When it is loss than [70 mai %], the transparency of resin and heat pokyvinył alcohol can be messured, for example based on JIS K 6726" polyvinyl alcohol test-method." than 30 moi % therefore, a saponification degree of the above-mentioned polyvinyl alochol has a 10039] An average degree of polymerization and a saponification degree of the above-mentioned exceeded, the reactivity of acetalization will fall, and moreover it will become easy to generate a block of resin during a reaction, and will become easy to follow difficulty on composition of resin. of resin layers with a parbon number enough by lass than three may not be obtained. If 10 is resistance may fell and reactivity may also fall it is a thing beyond 95 mol 5 more preferably.

40]kt is not limited especially as the above-mentioned aldehyde, but For example, propionaldehyde, czaldehyda, and a cinnamaldahyda, aromatio series, alicycle fellows aldehyde, etc. are mentioned utylaldehyde, isobutyraldehyde, a valeraldehyde, Aliphatic series, such as n~hexylaldehyde, 2~ mg, and n-buty/sidehyde of the carbon number 4 is excellent also in weatherability by use of yvinyt-apetal resin obtained and moreover becomes easy [manufacture of resin], it is more ylbutylaldehyde, and n-cotyl aldehyda. Since adhesive strength of each resin layer becomes ylbutydaldehyde, n~heotylaldehyde, n~ootyl aldehyde, n~nonyl aldehyde, n~decyl aldehyde, ferably, they are n-butylaidehyde of the carbon numbers 4-8, n-hexylaidehyde, 2ferred. These may be used alone and two or more sorts may be used together.

neter of potassium salt in an interlayer, it is preferred that it is 10 micrometers or less, and it is 5 rometers or less more preferably. The finer moderate still more desirable one of particle diameter arlayer is 10 micrometers or less, and it is 5 micrometers or less more preferably. About particle 41]In an interlayer of this invention, it is preferred that particle diameter of sodam saft in an

42]Since condensation of water may be promoted when particle diameter of the above-mentioned particle diameter of potassium saft, also in polyvinyf-acetal resin, since particle diameter may be lum selt exceeds 10 micrometers or particle diameter of potassium salt exceeds 10 micrometers, assium salt may decrease in process of film production, As for particle diameter of sodium salt an became a hig cause of a white blush mark by moisture absorption of an interlayer obtained. assium sait point out particle diameter in inside of an interlayer, Although particle diameter of 43]Aithough particle diameter of the above-mentioned sodium salt and particle diameter of lium salt in polyvinyl-acetal resin which is the main raw material, and particle diameter of 3, it is preferred that it is in a mentioned range.

44]Particle diameter of sodium sait in the above-mentioned interlayer and potassium sait can be asured by imaging of a secondary ion image which used a time-of-flight type secondary-ionss-spectroscopy device (TOF-SIMS).

ferably, sodium concentration is 0.5 ppm or more 15 ppm or less, and potassium concentration is 45]In an interlayer of this invention, it is preferred that sodium concentration is 50 ppm or less. sut patassium concentration in an interlayer, it is preferred that it is 100 ppm or less. More apm or more 100 ppm or less.

rdeyer exceeds 50 ppm and potassium contents exceed 100 ppm, e white blush mark may become lum element and a potassium element is visualized if a sodium content in the above-mentioned ning to be used, auch as water and raw material, by preparation of reain may be needed, great 46]Since it will grow up to be even a size by which a water molecule which gathered around a tarkable. Preparing an interlayer less than 0.5 ppm in any { of a sodium content in the aboventioned interlayer, and potassium contents] case, Treatment which lengthens dramatically a cess of washing a residual sodium element or a potassium element, or raises the degrees of e and expense may be required, and it may not be desirable practically.

47) Sodium concentration and potassium concentration in the above-mentioned interleyer can be me of a decomposition product with ultrapure water, it is the method of quantifying by the IOPintified by ICP luminescence ultimate analysis. After the above-mentioned ICP luminescence mete enelysis heats and decomposes a sample with suffuric soid and nitric acid and sets the

48] Mixing of the above-mentioned sodium and/or potassium, For example, in order to neutralize

radation of resin itself in a generation reaction of polyvinyl-acetal resin which is a process before oass can prevent an acid catalyst like indispensable chloride (HCI) remeining in resin, and causing acid catalyst of suffuric acid used for a reaction, obloride, etc. in preparation of polyvinyl-acetal 49]In a manufacturing method of the above-mentioned polyvinyi-acetal resin, a nautralization in, it originates in having used a neutralizer having contained a sodium element or potassium nents, such as sodium carbonate, sodium bicarbonate, sodium acetate, sodium hydroxide, assium carbonate, potassium bicarbonate, potassium acetate, and a potassium hydrate.

50] As the above-mentioned neutralizer, alkali metal salt and alkaline earth metal salt can be used. in if alkaline-earth metals remain so much in an interlayer unline an alkaline metal, they are

ferred at a point which can control a white blush mark under high humidity.

x//www4.jpdl.irpit.go.jp/cgi-bin/tren_web_ogi_ejje?atw_xrhttp\$3A%2F%2Fwww4.jpdli... 2009/06/15

JP,11-343152,A [DETAILED DESCRIPTION]

0051]As the above-mentioned alkeline earth metal sait, calcium sait, such as barium sait, calcium potassium salt, such as carboxylic acid and octylic acid, etc. as an adhesive strength regulator, a sodium element or a potassium element contains in usod water, raw material, especielly polyvinyl hydroxide, such as magnesium salt; barium hydroxide, such as aerated water matter magnesium, [0052]Wixing of the above-mentioned sodium and/or potassium, When adding sodium sait or magnesium hydroxide, and basic magnesium carbonate, etc. ere mentioned, for example, airohol, etc., and it happens also by a case where these remain in an interlayer.

10053]For example, an alkaline metal contained in the above-mentioned pure water can be reduced to polyvinyi albahol originates is sodium acatate generated in a manufacturing process of a polyvinyi alcohol raw material when saponifying polyvinyl acetata, and content is usually 0.4 to 1.5 % of the I ppm or less by using ion exchange water. On the other hand, an alkalina metal contained in

or less % of the weight, sodium elements with difficult washing contained in resin can be reduced. It is by rinsing polyvinyl-acetal resin, without performing the above-mentioned neutralization process, until [0055]]h a manufacturing method of the above-mentioned polyvinyl-acetal resin, it can also carry out to a white blush mark of a resin layer obtained can be held down to below the specified quantity until remains of an acid catalyst by [of 60 ** or less] drying at low temperature comparatively, corrosion it becomes plf five or more, and also drying below 60 **. By fully rinsing, alkali metal content leading [0054]Therefore, when content of sodium acetate uses a polyvinyl alcohol raw material which is 0.4 it becomes pH five or more. White preventing resin detenoration by mixing of an alkaline metal, and by ecid of a dryer can be prevented. Although a conventional method may be sufficient as a drying stabilized by washing strengthening etc., and a sodium element can be 50 ppm or lass. method, especially its vacuum-drying method is efficient, and excellent.

deterioration by mixing of an alkaline metal, or remains of an acid catalyst. At the time of washing, by efficiency by not less then 40 **. If wash water is lower than 40 **, resin will not fully swell but it will using 40-60 ** wash water preferably, resin in a stury swells, and acid (HGI) incorporated into resin water. It is for relising washing efficiency by temperature of water used at the time of washing being he hard to go up an effect. When wash water is more expersive than 80 **, while softening of resin not less than 40 ** paying attention to resin in a sturry swelling above 40 **, and preventing resin (0056) in the above-markioned washing process, it is preferred to wash with not less than 40 ** and its neutralized substance (alkaline metal inclusion) are flushed easily, and can raise washing may take place, particles may coalesce, formation of a block may be seen and a thing of stable particle diameter may not be obtained, improvement in a large affect cannot be expected as compared with 60 ** water, but it becomes useless also in energy.

chloride remover of a reaction using epoxide, and a method of periorming fiin formation can also be compounding polyvinyl-acetal resin, Polyvinyl-acetal resin can be obtained as a stop agent and a mentioned after that. It is expressed with following general formula (1) as the above-mentioned 10057]In order to prevent mixing of the above-mentioned sodium and potassium, carry out the acetalization reaction of polyvinyl alcohol, a chloride catalyst, and the aldehyde, and it faces epoxide, [0058]

Formula 1

3 R1-CH-CH-CH-13-CH--R7

3-epoxide, such as trimethylene oxide, a tetrahydrofuran, and tetrahydropyran, 1,5-epoxide besides 1,2-epoxide, etc. are mentioned — these — one sort — or two or more sorts may be used together. $3059 \mathrm{KR}^4$ and R^2 express hydrogen or an alkyl group.) n expresses the integer of 0–3. 1,4-epoxide, Especially as epoxide, ethyleneoxide, propylene oxide, etc. are preferred.

mentioned epoxide with the neutralizer of a chloride catelyst, and stopping an acetalization reaction 10060]As amount of the above-mentioned epoxide used, the effective dose which can perform stop of a reaction and chloride removal can be used. Degradation of ream by mixing of an alkaline metal and remains of an acid catalyst can be prevented by replacing the method of using the aboveusing epaxide, and also removing phloride.

.0061]In this invention, as for an interlayer for glass laminates, in order to prevent a white blush mark

http://www.fpdl.inpit.go.jp/cgi=bin/fran_web_cgi_ejje?atw_u=https%34%2F%2Fwww4.pdl... 2009/05/15

mound which can form sodium salt and potassium salt, and a complex, resin, and a plasticizer as 82] Amine which is compatible in organic acid, resin, and a pleaticizer which are compatible in a above-mentioned dispersing agent is mentioned.

83] The compound which can form the above-mentioned sodium saft and putassium sait, and a wermentioned polyvinyl-acetal resin absorbs moisture, in order to make it hard to approach in opex can control a white blush mark of an interlayer for giass faminates obtained, even if the er by carrying out hydraphobing of the circumference of metal salt, such as sodium salt and

assium saft, and a complex. For example, ethylenediaminetebraecetic acid, salicyladdeltyde, salicylic ethylgtyoxime, 1,1-cyclohexane 2 acetic acid, salicylalduxime, a glycine, ets. are mentioned. These 64]It is not limited especially as a compound which can form the above-mentioned codeum and 1. Salicylamilide, oxalic acid, a 1,10-phenanthroline, an acetylacetone, 8-hydroxyquinoline, be used alone and may use two or more sorts together.

in, it is preferred that they are 0.02 - the amount part of duplexs to the polyvinyl-acetal resin 100 ve-montioned weight section. If a preventive effect of a white blush mark by moisture absorption assium salt, and a complex is based on quantity of metal saft which remains to pelyvinyl-acetal eeded, compatibility with the above-mentioned polyvinyl-acetal resin may become poor, and a , become insufficient if it is less than 0.02 weight sections and the amount part of duplexs is 65]Atthough an addition of a compound which can form the above-mentioned sodium and blom may axise in transparency. It is 0.05 to 1 weight section more preferably,

88]As the above-mentioned dispersing agent, amine which is compatible in organic acid, resin, and lesticizer which are compatible in resin and a plasticizer can also be used. As organic acid which is 67]R is using at least one sort more preferably from a group which consists of suffonic acid whose apatible in the above—mentioned resin and a plasticizor, polymer acid, such as monomer acid, such suffenie acid, carbaxylic acid, phospheric acid, and nitric acid, polysulfone acid, and pelycarboxylic bon numbers are 2-21, carboxylic acid whose carbon numbers are 2-20, and phusphoric acid eto, are mentioned, and suffonic acid, carboxylic acid, and phosphoric acid are especially ferred. These may be used independently and may be used together two or more sorts. ntioned resin and a plasticizer.

ressed with following general formula (II) as organic acid which is compatible in the above-

rophobicity, compatibility with polyvinyl-acetal resin worsens, and there is a possibility of causing nbers are 1–18, or the aromatic system hydrocarbon group whose carbon numbers are 1–18.) \mathbb{R}^4 $59
m KR^3$ expresses among a formula the aliphatic series system hydrocarbon group whose carbon 70]In the suitoric acid whose above-mentioned carbon numbers are 2-21, if a carbon number is resses a hydrogen atom, the aliphatic series system hydrocarbon group whose cerbon numbers than two, When hydrophills nature may become high, compatibility with polyvinyl-acetal resin versen, distributing may become insufficient and a carbon number exceeds 21, it becomes 1--18, or the aromable system hydrocarbon group whose carbon numbers are 1--18. se separation. A carbon number is a thing of 7-18 more preferably.

hatic series system, the thing of an aromatic system, etc. The above-mentioned carbon number is limited especially as sulfonic acid which is 2-21. For example, benzenesulfonic acid, naphthalene 73]As sulfanic acid which is 2-21, the above-mentioned carbon number can use the thing of an onic acid, alkyi sulfenic acid whose carbon numbers of an alkyl group are 2~21. The carbon ther of an alkyl group is alkyl naphthalene sulforio acid etc. which are 2-11, and the carbon ther of the alkylbenzene sulfonic acid which is 2-15, and an alkyl group specifically, P- :://www4.indi.inpit.go.jn/cgi-bin/tran.meb.cg.ejje?etw.u=http%3A%2F%2F%yew4.jpdji... 2009/06/15

JP,11-343152,A [DETAILED DESCRIPTION]

acid, mestrylene sulfonic acid, etc. are mentioned. These may be used sione and may use two or more tolusnesulfonio acid, dodecylbenzenesulfonio acid, camphor sulfonio anld, hydroxypropanesulfonio

part of duplexs, degradation of resin may be promoted or this suffenic acid itself may cause a white than 0.01 weight sections to moisture absorption may become insufficient and exceeds the amount mentioned weight section. If a preventive effect of a white blush mark according that they are less preferred that they are 0.01 – the amount part of duplexs to the polyvinyl-acetal resin 100 above-10072] As for an addition of sulfonic acid whose above-mentioned carbon numbers are 2-21, it is blush mark. It is 0.03 to 1 weight section more preferably.

hydrophobicity, compatibility with polyvinyl-acetal resin worsens, and there is a possibility of causing (0073) in carboxylic acid of 2-20 the above-mentioned carbon number. When hydrophilic nature may become it high that a carbon number is less than two, compatibility with polyvinyl-acetal resin may worsen, distributing may become insufficient and a carbon number exceeds 20, it becomes phase separation. A carbon number is a thing of 6-14 more preferably.

acid, pimelio scid, sebacic scid, oleie acid, benzoic scid, tollic scid, napitthois scid, 1,1-cyclohexane 2 (0074] As carboxylic acid which is 2-20, the above-mentioned carbon number can use a thing of an ethylhexyl acid, lauric acid, myristic acid, stearic acid, oxalic acid, malonic acid, suocinic acid, adiple aliphatic series system, a thing of an aromatic system, etc. It may be dicarboxyiic acid. The aboye acetic acid, salicylic acid, etc. are mentioned. These may be used alone and may use two or more mentioned carbon number is not limited especially as carboxylic acid which is 2-20. For example, acetic acid, propionic acid, butanoic acid, isobutyric acid, 2-ethylbutanoic acid, Octanoic acid, 2-

preferred that it is 0.01 to 3 weight section to the polyvinyl-acetal resin 100 above-mentioned weight section. When a preventive effect of a white blush mark according that they are less than 0.01 weight [0075] As for an addition of carboxylic acid whose above-mentioned carbon numbers are 2-20, it is compatibility with resin becomes proor and transparency has a possibility of a problem arising or sections to moisture absorption may become insufficient and exceeds three weight sections. promoting degradation of resin. It is 0.05 to 1 weight section more preferably,

hydrophobicity and compatibility with polyvinyl-acetal resin may worsen. Carbon numbers are 6-12 formula (II), when a carbon number of the above-mentioned alighatic series system hydrocarbon group or the above-mentioned aromatic system hydrocarbon group exceeds 18, it may become 10076] In R³ and R⁴ of phosphoric acid which are expressed with the above-mentioned general

expressed, it is not limited, but can use phosphoric acid generally used, and specifically, For example, methylphosphoric acid, ethylphosphoric acid, propylphosphoric acid, isopropylphosphoric acid, Butyl diethylphosphoric acid, diisoprapylphosphoric acid, dioctylphosphoric acid, diphenylphosphoric acid, phosphate, laurylphosphate, stearylphosphoric acid, 2-ethylhexyl phosphoric acid, Di(2-ethylhexyl) dibenzylphosphorio acid, etc. are mentioned. These may be used alone and may use two or more 10077] Especially as phosphoric sold which is the above-mentioned general formula (II) and is phosphoric acid, isodecyl phosphoric acid, phenylphosphoric acid, dimethylphosphoric acid,

[0078]As for an addition of phosphorio and expressed with the above-mentioned general formula (II), above-mentioned weight section. If a preventive effect of a white blush mark by moisture absorption may become insufficient when it is less than 0.01 weight sections, and the amount part of duplexs is exceeded, degradation of reain may be promoted or this phosphoric acid itself may cause a white it is preferred that they are 0.01 - the amount part of duplexs to the polyvinyl-acetal resin 100 blush mark. It is 0.03 to 1 weight section more preferably.

general formula (III) can be conveniently used for amine which is compatible in the above-mentioned together with amine which is compatible in resin and a plasticizer. What is expressed with following 19079] Organic acid which is compatible in the above-mentioned resin and a plasticizer is used esin and a plasticizer

Formula 33



81](R⁵, R⁶, and R⁷ are a hydrogen atom, an alighatic series system hydrocarbon group whose carbon numbers are 1–20 on an aromatic system hydrocarbon group whose carbon numbers are 1–20 on general aromatic system hydrocarbon group or allower narrows aromatic system hydrocarbon group or above-mentioned alighatic series system hydrocarbon group or above-mentioned aromatic system hydrocarbon group are of R⁵, it may become hydrophobicity of compatibility with polyvinyl-scetal resin may worsen. As for any one of R⁵, R⁵, and R⁷, it is formed that it is a long chein, and a hydrocarbon group and R⁷ R⁵ and whose R⁶ are a hydrogen m and the carbon numbers 1–2 are a hydrocarbon group of the carbon numbers 6–16 mere ferably.

ylamine, propylamine, hexylamine, octylamine. Decyl amine, dodocyl amine, tetradecylamine, aniline. 84]As for an addition of amine expressed with the above-mentioned general formula (III), when a sticizer, it is preferred that they are 0.01 - the amount part of duplexs to polyvinyl-acetal resin tion number uses suffortic sold which is 2~21 as organic sold which is compatible in resin and a stioned by nitrogen-containing fieterocyclic compounds, such as aromatic amines, such as 1, 2 ne, such as dipropyl amine, dihexyl amine, diootyl amine, and M-methylaniline, Trimethylamine, akidine. The 1st class amina, such as naphthylamine, Dimethylamine, diethylamine, Sacondary 83]As amine expressed with the above-mentioned general formula (III), For example, they are ome insufficient when it is less than 0.01 weight sections, and the amount part of duplexs is I weight section. When a preventive offect of a white blush mark by moisture absorption may tiery amine, such as triethylamine, M.N-dimethylhexylamine, N.N-dimethyl octylamine, M.Needed, compatibility with resin becomes paor, a problem may arise in transparency or an alkylamine of the 3rd class, and aniline, and pyridine, etc., and specifically, Methylamine, ethyldecyj amine, M.N-dimethyldedecył amine, N.N-dimethylaniline, and pyridine, etc. are ntioned. These may be used alone and may use two or more sorts together. rlayer may color it it. It is 0.02 to 1 weight section more preferably.

85]As for an addition of amine expressed with general formula (III), when a carbon number uses boxytic acld which is 2-20 as organic acid which is compatible in the above-mentioned resin and a sticizer, it is preferred that it is 0.01 to 3 weight section to polyvinyl-acetal resin 100 weight. Sion, When a preventive effect of a white blash mark by moisture absorption may become ifficient if it is lass than 0.01 weight sections and three weight sections are exceeded, neetingly with resin becomes poor, a problem may arise in transparency or an interlayer may color it is 0.05 to 1 weight section maye preferably.

88] Amina which is compatible in organic acid, resin, and a plasticizer which are compatible in resin tal ion which constitutes this metal salt, and its countar ion, or [and / that metal sait, which these combined by kneading resin at the time of film production is distributed in resin, and metal salt of lexs to polyvinyl-acetal resin 100 weight section. When a preventive effect of a white blush mark ucture and a molecular weight suitably and to be used according to the degree of acetalization of l a plasticizer which are used as the above-mentioned dispersing agent, Respectively it becomes densation of water is controlled and polyvinyl-acetal resin absorbs moisture, a white blush mark ressed with the above-mentioned general formula (II) as organic acid which is compatible in the ount part of duplexs is exceeded, compatibility with resin becomes poor, a problem may arise in 86]As for an addition of amine expressed with general formula (III), when using phosphoric acid moisture absorption may become insufficient when it is less than 0.01 weight sections, and the face of metal salt of particle state which exists in polyvinyl-acetal resin, and combines with a such as sufform? Ion, a carboxyl ion, phospheric acid ion, and AMMONIUM ion, it acts on the we mentioned resin and a plasticizer, it is preferred that they are 0.01 - the amount part of 87]As for the above-mentioned organic acid or amine, it is preferred to choose melecular tide state becomes small as the result.] — or it disappears. For this reason, even if local isparency or an interlayer may color it it is 0.05 to 1 weight section more preferably. above-mentioned polyvinyl-acetal resin or a kind of the above-mentioned plesticizer.

JP,11-343152,A [DETAILED DESCRIPTION]

of an interlayer for glass laminates obtained can be controlled. Among organic acid which is competible in resin and a plasticizer which are used as the above-mentioned dispersing agent, especially in phosphoric acid, since a role of a coupling agent of resin and glass is also played, and it has the character in which a film and glass do not exfoliate easily even if it absorbs moisture, exfoliation by moisture absorption of a glass laminate periphery can be controlled.

[0089]As for an interlayer for glass laminates, in this invention, it is preferred to contain at least one sort chosen from a group which consists of alkeli metal selt and elkeline earth metal salt as an adhesive strength regulator.

[0090]It is not limited especially as the above-mentioned alkali metal saft and alkaline earth metal saft, for example, safts, such as potassium, sodium, and magnesium, are mentioned. As the above-mentioned saft, inorganic acid, such as organic acid, chlorides, such as carboxylic acid, such as octylic acid, hexylacid, butanoic acid, acetic acid, and formic acid, and nitric acid is mentioned. [0091]As the above-mentioned alkali metal saft and alkaline earth metal saft, it is more preferred that they are akkali metal sait of organic acid of the carbon numbers 5-16 and the alkaline earth metal saft of organic acid of the carbon numbers 5-16 and the alkaline earth metal saft of organic acid of the carbon numbers 5-16, it is magnesium saft of carboxylic acid of the carbon numbers 6-16, or dicarboxylic acid preferably.

10092]It is not limited especially as magnesium sait of the above-mentioned carboxylic acid or dicarboxylic acid, For example, 2-ethylbutanoic acid magnesium, valeric-acid magnesium, hexanoic acid magnesium, magnesium nonanoste, decanoic acid magnesium, magnesium nonanoste, decanoic acid magnesium, gutaric acid magnesium, acipic acid magnesium, acid magnesium, gutaric acid magnesium, acid magnesium, acid magnesium, acid magnesium, acid magnesium, acid magnesium, gutaric acid acid magnesium, acid magnesium, acid magnesium, acid magnesium, acid carboxylic acid of the above-mentionied carbon numbers 6-10, or dicarboxylic acid, 20 existing in a form of a sait, without ionizing in a film, and drawing a water molecule near, it is thought that it is possible to stop adhesive strength between an interlayer and glass. and penetration-proof performance of a glass laminate obtained by this can be made good. Since it is distributed ever a membrane surface at high concentration, without condensing in a flim and an excessive white blush mark at the time of moisture absorption is not caused while a small quantity shows an adhesive strength adjustment effect, it is desirable.

[0094]As for the above-mentioned alkali metal salt and alkaline earth metal salt, it is preferred that particle diameter is 3 micrometers or less, and they are 1 micrometer or less more preferably. Since it will grow up to be even a size by which a water molecule which gathered around alkali metal salt, and/or alkeline earth metal salt is visualized if it exceeds 3 micrometers, a white blush mark may become remarkable and may not be preferred.

[0095]It is not limited especially as a means for the above-mentioned particle diameter to be 3 micrometers or less. For example, even if it is a method of using a compound which is easy to dissolve in polyvinyl-acetal resin or a plasticizer as an adhesive strength regulator, and a compound which is hard to dissolve in polyvinyl-acetal resin or a plasticizer, A method of using what it is hard to condense in polyvinyl-acetal resin and a plasticizer, a method of using together a dispersing egent which distributes them, a compatibilizer, etc., etc., are mentioned.

[0096]As a compound which is easy to dissolve in the above-mentioned combination, polyvinyl butyral resin is used as polyvinyl-acetal resin, for example, In combination using triethylene glycol 2-ethyl butyrate as a plasticizer, organic acid selt, such as octanoic acid magnesium, neo decanoic acid magnesium, and adipic acid magnesium, etc, are mentioned, and these one sort or two sorts or more are used suitably.

[0087]As a potessium compound which is easy to dissolve in the above-mentioned combination, patassium salt of organic acid, such as cetanoic acid potassium, neo decancic acid potassium, and potassium stearste, etc. are mentioned, and these one sort or two sorts or more are used suitably. [0098]As a sodium compound which is easy to dissolve in the above-mentioned combination, sadium salt of organic acid, such as sodium cotanoste, neo decane sodium, and sodium stearate, etc. are mentioned, and these one sort or two sorts or more are used suitably.

[0088]As a compound which is daring combination of what it is hard to dissolve in the abovermentioned combination, and is hard to condense, magnesium salt of inorganic acid, such as a magnesium chloride and a magnesium nitrato, etc. are mentioned, and these one sort or two sorts or more are used suitably, for example.

[0100]As a disparsing egent which may be distributed according to concomitant use, or a competibilizer, a compound which is hard to dissolve in the above-mentioned combination, Although

thtp://wwwf.ipdlinpit.go.jp/cgi-bin/tran.wsb_cgi_ejje?atw_u=littp%3A%2F%2Fwwwf.ipdli... 2009/08/15

limited in particular, long chain system organic acid, such as alcohols and octanoic ecid, such as and and octyl alcohol, and nonancic acid, is mentioned, and these one sort or two sorts or more used suitably, for example,

spound which it ranks second and is hard to condense in polyvinyl-acetal resin and a plasticizer is theizer also in various above-mentioned methods is the most preferred, and a method of using a 01]A method of using a compound which itself tends to dissolve in polyvinyr-acetal resin or a

acture of a diester system compound. Since it can distribute stably and uniformly and can exist in in by having a structure similar to an acid component of a diester system compound used as a 02]As the above-mentioned alkeli metal salt and alkaline earth metal salt, when using a diester tem compound as a plasticizer, it is preferred that it is what has an acid component and the sticizer, eging is not caused.

of the carbon number 5 or carboxystic acid of 8 contain as an adhesive strength regulator, Since a ig hereafter called "3GH") or a dihexyl herse mackerel peat (henceforth "DHA"), by making metal nosforth "3GO") as a plasticizer for same reason, it is preferred that metal salt of carboxylic acid 03]As the above-mentioned plastinizer, triethylene glycol di-2-ethyl butyrate. When using (it also vention from a white blush mark and prevention from a fall with the passage of time of adhesive of adhesive strength of an interlayer and glass with the passage of time can be prevented and ength can be reconciled, it is desirable. When using triethylene glycol di-2-ethylhexamoate he carbon numbers 6-8 contains. When using tetraethylene Glico Lod'z 2-ethylhexanoate nceforth "4GO") as a plasticizer, it is preferred that metal salt of the carbon number 8 or boxylic acid of 7 contains.

ad di-heptanoate (3G7) or tetraethylene glycol di-heptanoate (4G7), it is preferred to use a side 04]In order to prevent hydrolysis by heat at the time of film production of polyvinyl-acetal resin sticized [above-mentioned] as much as possible, it compares with a plasticizer like tristhylene in type plasticizer fike 3GH which cannot cause hydrolysis easily, 3GO, and 4GO, or a horse skerel peat system type plasticizer like DHA.

antage of being hard to cause hydrolysis, as compared with 3G7 and 4G7 grade which an organic npared with 3GH, since the above-mentioned 3GO or 4GO are high boiling points, they have the we-mentioned 3GH, 3GO, 4GO, and DHA. Since the characteristic which above-mentioned 3GH 06]Above-mentioned 3GH, 3GO, 4GO, and DHA may use together with other plasticizers which 4GO, and DHA have can weaken with other plasticizers if it is 50 % of the weight or more, an I ingredient of a raw material is a side chain type, and are straight chain types. For example as 05]Above-mentioned 3GH has a track record long as a plasticizer for interlayers, and has the ntioned 3GH, 3GO, 4GO and DHA, and other plasticizers is not limited, it is preferred that the ount of concomitant use of other plasticiners is less than 50% of the weight of a plasticizer of y be used alone and mentioned later. Although a concomitant use rate in particular of aboveset of an adhesive strength regulator used corresponding to these may not no longer be antage of being hard to vaporize at the time of linn production or doubling processing.

f metal saft (carbon number 5), haxanoic acid (2-ethylbutanoic acid) metal saft (carbon number 6), we-mentioned plasticizer. The aboye-mantioned carboxylic acid may be a straight chain type, and 07]Matal saft of carboxylic ecid as an adhesive strength regulator used when setting a plasticizer he above-mentioned interlayer as a specific thing, Although not limited in particular, pentanoic standio acid metal salt (carbon number 7), octandio acid metal salt (carbon number 8), etc. are ntioned, and these one sort or two sorts or more are suitably used according to a kind of the y be a side oftain type.

08]The moisture resistance of an interlayer obtained may become insufficient in case of metal salt passage of time may become insufficient conversely in case of metal salt whose carbon number use carbon number of the above-mentioned carboxylic acid is too small, generating of elbinism y become large, and a fall preventive effect of adhesive strength of an interlayer and glass with parboxylic acid is too large.

00]Metal salt of carboxylic acid as the above-mentioned adhesive strength regulator, Although it. f be used independently, respectively, formic acid magnesium, magnesium acetate. A metal salt tem adhesive strength regulator of carboxylic acid of the carbon numbers 1-4 like magnesium n//www4jpdiinpit.gojp/cgi-bin/tran_web_cgi_cjie?atw_u=http%3A%2F%2Fwwn4jpdii... 2009/06/15

JP,11-343152,A [DETAILED DESORIPTION]

adhesive strength regulator, the addition has 0.01 to 0.2 preferred weight section to polyviny-acetal resin 100 weight section. Since an adhesive strangth adjustment effect will be lost if it is less than regulators, such as a denaturation silicone oil system adhesiya strength regulator mantioned later. carrying out blead out, there is a possibility that adheave strength of an interlayer and glass may 0.01 weight sections, penetration–proof performance of a glass laminate obtained may fall, If 0.2 0110]When adding the above-mentioned alkali metal salt and/or alkaline earth metal salt as an weight sections are exceeded, while spoiling the transparency of a glass faminate produced by propanoate and magnesium butanoate; it may be used together with other adhesive strongth decline too much. It is 0.03 to 0.08 weight section more preferably.

[0111]As for sodium concentration, since especially a white base mark happens easily when the above-mentioned alkali metal salt is sodium sait, being set to 50 ppm or less is preferred. As for potassium element concentration, since a white blush mark happens casily also when the above~ [0112]The above-mentioned alkali metal salt and/or alkaline earth metal salt, Sulfurio acid used mentioned alkali metal saft is potassium saft, being set to 100 ppm or less is preferred.

earth metal salt as a neutralizer of an acid catalyst of chloride etc., and a reaction of polyvinyl-acetal polyvinyl-acetal resin. The above-mentioned metal salt may have contained in various raw materials. cothers / in a case of adding as the above-mentioned adhesive strength regulator } in a reaction of water, etc. which were used in a case where it originates in having used alkali metal sait and alkaline resin. Alkali metal salt and alkaline earth metal salt as the above-mentioned neutralizer can be diverted to some other purpose as an adhesive strongth regulator.

[0113]An interlayer for glass laminates of this invention consists of a plastic resin film which consists of additive egents, such as an above-mentioned dispersing agent and an adhesive strength regulator, by the above-mentioned polyninyi-acetal rasin, a plasticizer, and necessity.

[0114] As a plasticizer used in this invention, phosphoric acid system plasticizers, such as organic ester system plasticizers, such as a publicly known plasticizer used for this kind of interlayer, for example, menobasic acid ester, and polybasic acid ester, an organic phosphorus acid system, an organio phosphorous acid system, etc. are used.

ester obtained by a reaction with organic acid, such as butaneic acid, isobutyric acid, caproic acid, 2ethylbutsnoid adid, hentanoid adid, n-odtylio adid, 2-ethylhexyl adid, pelargonio adid (n-nenylio adid), [0115] In the above-mentioned monobasic acid ester, for example Triethylene glycol, Glycol system and decylacid, is preferred. In addition, ester of tetraethylene glycol, tripropylene glycol, and the above-mentioned organic acid is also used,

sebacic acid, and axelaic acid, and straight chain shape of the carbon numbers 4-8 or branched state [U116] As the shove-mentioned polybasic acid ester, ester of organic acid, such as adipic acid. alcohol is preferred, for example.

propylene glycol di-2-ethyl butyrate, 1,4-butylene GURIKORUJI 2-ethyl butyrate, 1,2-butylene-glycol [0119]As the above-mentioned phosphoric acid system plasticizer, tributoxyethyl phosphete, isodocyl compound which consists of dicarboxylic ecid and monohydric elochol, or consists of monocarboxylic [0117] As an example of the above-mentioned organic ester system plasticizer, Triethylene glycol diphonyl phosphate, triisopropyl phospitite, etc. are preferred. It is preferred to blend a diester system [0118]in addition, ethylene glycol di-2-ethyl butnate, 1,3-propylene stycol di-2-ethyl butyrste, 1,4di-2-ethylene butyrate, disthylene-glycol di-2-ethyl butyrate, Diethylene-glycol di-2-ethylhexoate, glycol di-n-actoate, Triethylene glycol di-n-HEPUTOETO, tetraethylene glycol di-n-HEPUTOETO, other dividy sebacate, dioctylazelate, and a dibutylearbitol horse mackerel peat are used suitably. dipropylene giyool di-2-ethyl butyrate. Triethylene giyool di-2-ECHIRUPENTOETO, tetraethylene 2-ethyl butyrate, triethylene glycol di-2-ethylbaxoate, Triethylene glycol dicaprylate, friethylene glycol di-2-ethyl butyrate, dethylene-glycoi JIKAPURIETO, etc. are used as a plasticizer. acid and dihydric alcehol in the above-mentioned plasticizer.

penatration resistance of a glass faminate which will be obtained if it is less than 20 weight soctions iaminates, such as conversion silicona oil for controlling penetration resistance besides a dispersing may falt. if 70 weight sections are exceeded, a plasticiter will carry out bleading, optical strain may polyviny?-acetal resin 100 weight section, and it is 40 to 60 weight section more preferably. The [0120]As the above-mentioned amount of plasticitiers, 20 to 70 weight section is preferred to (01213) this invention, a publicly known additive agent currently used for interlayers for glass become large or the transparency of a resin layer and an adhesive property may fall

int and an adhesive strength regulator, an ultraviolet ray absorbent, light stabilizer, an antioxidant, urface-active agent, and colorant, as an additive agent can be blended.

diffed silicone of, for example, is indicated by JP,55-29950,B. Ether denaturation silicone oil, ester aturation silicone oil, and amine denaturation silicone oil carboxyl modified silicone oil etc. are 22]An epoxy deneturation silicone oil which is not limited especially as the above-mentioned ntioned. These modified silicane oil is fullds produced by making a compound which should saturalize to a polysiloxane react generally.

23]In this invention, it is following general formula (IV).

25]Epoxy modified silicone oil, following general formula (V) which are expressed with (I and m ress 30 or less positive integer among a formula)

27]Ether modified silicone all expressed with (x and y to which I and an express 30 or less positive secr express 20 or less positive integer among a formula), and following general formula (VI)

$$CH_{\delta} = CH_{\delta} + CH_{\delta} + CH_{\delta} + CH_{\delta} + CH_{\delta}$$

$$CH_{\delta} = CH_{\delta} + CH_{\delta} + CH_{\delta} + CH_{\delta}$$

$$CH_{\delta} = CH_{\delta} + CH_{\delta} + CH_{\delta} + CH_{\delta}$$

$$CH_{\delta} = CH_{\delta} + CH_{\delta} + CH_{\delta} + CH_{\delta}$$

29]Ester modified silicane oil expressed with it and m express 30 or less positive integer among a Johner, in this invention, what is expressed with a structural formula of a random copolymer is mula) is used especially preferably. Although each modified silicane oil shown by the above~ ntioned general formula (IV), (V), and (VI) is expressed with a structural formula of a block d similarly.

30]The above-mantioned modified silicone oil may be used alone, and may use two or more sorts ether. As for a molecular weight of the above-mentioned modified silicone oil, 800–5000 are ferred. If localization to the surface falls that it is less than 800 and 5000 is exceeded.

npatibility with resin becomes poor, bleed out will be carried out to a membrane surface, and asive strength with glass will fall to it. It is 1500-4000 more preferably.

will be carried out to a membrane surface, and adhesive strength with glass will fall to it. It is 0.03 then to the polyvinyl-scetal resin 100 above-mentioned weight section. If a preventive effect of a somes insufficient and exceeds 0.2 weight section, compatibility with resin becomes poor, bleed 31]An addition of the above-mentioned modified silicone oil has 0.01 to 0.2 preferred weight. te blush mark according that they are less than 0.07 weight sections to moisture absorption

32]It is not limited especially as the above-mentioned articuldant, but as a thing of a phenol 0.1 weight section more preferably.

p//www4.indl.impit.go.jp/ogi-bin/tran.web_agi.ejje?atw.c=http%3A%2F%2Fwww4.ipdl.i... 2009/06/15

JP,11-343152,A [DETAILED DESCRIPTION]

Chemical Co., Ltd.), Tetrakis [methylene-3-(3'-5'-di-t-butyl-4'-hydroxyphenyl) propionate] methane system, For example, t-butyflydroxytoivene (BHT) ("Sumi Rider BHT (trade name)" by Sumitemo (IRUGA NOx 1010, Ciba-Gaigy make) etc. are mentioned.

0133It is not limited especially as the above-mentioned ultraviolet ray absorbant, but as a thing of a Gtha-Geigy make), A thing of hindered amine systems, such as thing.LA-57 (made by ADEKAAGASU) benzotríazol system, for example, 2-(2'-flydroxy-5'-methylphenyl) benzotríazol (the tínuvín P.) The Geigy make), A 2-(2"-hydroxy-3"-t-butyl-5"-methylpinenyl)-5-chlorobenzotriazole (the tinuvin 326, of benzatriazoi systems, such as 2-(2"-hydroxy-3' and 5"-di-amyl phonyl) benzotriazoi (the timiyin Oba–Geigy make, 2–(2'–hydroxy–3' and 5'–di–t–buthylphenyi) benzotriazol (the tinuvin 320, Oba– 328, Olba-Geigy make), etc. are mentioned.

ADEKA stub LA-57" by the Asahi electrification company etc., (trade name) is mentioned. As the above-mentioned surface-active agent, sodium lauryl suffate, alkylbenzene sulfonic acid, etc. are [0134]As the above-mentioned light stabilizer, a thing of a hindered amine system, for example,

extrusion method, the calendar method, the pressing method, casting method, a tubular film process. plasticizer and if needed] for the specified quantity, A film is produced to a sheet shaped with an 10135]A manufacturing method of an interlayer for glass laminates of this invention. Although not etc., and a resin layer is fabricated, and let this be an interlayer, after kneading this uniformly. limited in particular, other additive agents are blanded with each above-mentioned resin [a

the minimum --- when required penetration resistance and weatherability are taken into consideration, [0138] thickness of the whole interleyer for glass laminates of this invention --- as a glass laminate generally the range of 0.3~1.6 mm is preferred like thickness in the usual interlayer for glass

[8137] As a glass plate used for the above-mentioned glass laminate, not only an inorganic clear glass imard but orgenio clear glass boards, such as a polycarbonate board and a polymethylmethacrylate

glass, sheet glass containing a line, heat ray absorption plate glass, and colored sheet glass, etn may be mentioned these may be used independently, and two or more sorts may be used together. An norganio clear glass board and an organic clear glass board may be laminated. Thickness of glass limited, Various inorganic glass, such as float sheet glass, polished plate glass, figured glass, wire [0138]Especially as a kind of the above-mentioned inerganic clear glass board, it is not what is should just be suitably chosen by use and is not restricted in particular by it.

[0139]In order to manufacture a glass laminate of this invention, a process of the usual glass laminate consists of a resin layer fabricated by an above-mentioned method, and putting this into a rubber bag is adopted. For example, between transparent glass plates of two sheets, pinch an interlayer which and carrying out decompression sucking at about 70-110 **, carry out preliminary adhesion and it. ranks second, it is manufactured by performing this adhesion by a pressure of about ten to 15

kg/om² at about 120-150 ** using a press, using autoclave.

temperature of 60-100 ** at the same time it makes the above-mentioned interlayer which comes at interlayer / glass plate into a rubber bag, and more specifically for example, in autoclave, –For [10 to 30 minutes] grade heat crimping is carried out by a pressure about temperature of about 60-100 **, mentioned above in this manufacturing method, and setting up suitably terms and canditions, such as earrying out suction deseration, by within the limits about the above, it can adjust so thet it may fall least to produce a film polyvinyl butyral resin plasticized between glass plates of a couple intervene and carries out suction descration under decompression. Put a layered product of a glass plate / a sticking-by-pressure pressure, sticking-by-pressure time, and a decompression degree when and 1 – 10 kg/cm², carrying out suction deseration under decompression of an about [500-[0141] By limiting temperature when carrying out heat crimping to the range of 60-100 ** as [0140]In a manufauturing method of a glass laminate, heat crimping may be carried out at 180mmilg], and it carries out by performing deseration and adhesion simultaneously within the fitness range of a request of adhesive strangth of an interlayer and glass.

[Example]Although an example is fung up over below and this invention is explained to it in more detail, this invention is not limited only to these examples. The "part" in an example means a "weight

http://wwwf.ipdl.inpit.go.jp/cgi-bin/tran.web.cgi.ejie?atw.u=http%3A%2F%2Fwww4.ipdi1... 2009/06/15

JP,11-343152,A [DETAILED DESCRIPTION]

npiete, superfluous water washes, and unreacted n-butylaldehyde is flushed. The sodium hydroxide 43]adding the average degree of polymerization 1700, and the degrees of polyviny! alcohol 275g of issolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride alysts [201 g of] and the orbutylaideliyde 148g were added, this temperature was held, and the erfluous water. The vinyi-acetal ingredient (degree of acetalization) of this resin was 65.0-mal %, onification of 98.8 mol to the preparation pure water 2890g of Example 1 (1) resin --- warming ctant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction ttion which is a general-purpose neutralizer neutralized the chloride catalyst, and also white võered polyvinyi butyral resin was obteined shrough rinsing and desiccetion for 2 ficurs with the vinyl acetate component was 1.1-mol %.

rying out melt kneading of this with a roll mill, with the press—forming machine, press forming was 44](2) Manufacturs above-mentioned polyvinyl-butyrel-resin 100 weight section of an interlayer, rometer and potassium salt of the particle diameter of the sodium salt in an inberlayer was less ging of the secondary ion image using a time-of-flight type secondary-ion-mass-spectroscopy ried out for 30 minutes, and 150 ** of 0.76-mm-thick interlayers were obtained. The result of ame of this decomposition product with ultrapure water, it is the method of quantifying by the F-SIMS) device (product [made by PHEVANS] TFS-2000 type). The particle diameter of 1 ing measured the particle diameter of the sodium salt in an interlayer, and potassium salt by mate analysis heats and decomposes a sample with sulfunc acid and nitric acid and sets the n 0.5 micromater. The bottom was a result and 8 ppm in fixed quantity in ICP luminescence er mixing triethylane-glycol-di-2-ethyl-butyrate 40 weight section as a plasticizer and fully mate analysis about the sodium element content of this interlayer. After ICP luminescence "-AES method using an IOP-AES device (the "IOAP-575 type" by a depanase jar rel ash

utes at 86 more **. Thus, this adhesion was performed for the glass laminate by which preliminary osion was carried out for 20 minutes on prossure 12 kg/cm² and conditions with a temperature of rubber bag and parrying out indirect dessifurization mind with the degree of vacuum of 20torr for minutes, it moved to 90 4× over, deaerated, and the vaccum press was carried out, holding for 30 se faminate, an adhesive examination (pan Meru examination) and the moisture-proof white-blushinate with a transparent float glass (30 cm by 30 cmx2.5 mm in thickness), After putting this in in i ** in air type autoclave, and the transparent glass laminete was obtained. About the obtained 45)(3) it isserts from both sides using the manufacture above-mentioned interlayer of a glass k sex test were done by the following methods.

46]A quality assessment (1) adhesive-property (pen Meru) examination glass laminate is neglected the which carried out grade attachment beforehand, as shown in Table 1. This is for the adhesive adjusted to -18 ****0.8 ** temperature, and this is struck with the hammer whose weight of a ree of exposure of the film after glass carries out partial avuision is judged with the boundary id is 0.45 kg, and it grinds until the particle diameter of glass is set to 6 mm or less. And the ongth of a glass plate and an interlayer to judge whether it is in the predetermined range.

ble 1

後にメババ çs ¢: ** ur v۶ ಭಾ 対数の数点数(米) 2.23.75 :e: ∞ 0 (C) क १८३ <u>ئ</u> ج 0 23 226 Ġ

p.//www4.indl.inpit.go.jp/cgi-bin/tran_web_cgi_ejie?atw_c=http%3A%2F%2Fwww4.ipdli... 2009/06/15

[0148](2) The moisture-proof white-blush-mark sex-test resin layer was out into the size of 4x4 cm. after was measured using the integral equation turbidity meter (made by Tokyo Denshoku Co., Ltd.). it was immersed in the fon exchange water of ordinary temperature (23 **), and Hayes of 24 hours The result was shown in Table 2.

10149]In preparation of resin of example 2 Example 1, washing and a washing process after putting in for the sodium element content in the obtained interlayer, the particle diameter of 3 micrometers and changed washing and a washing pracess after putting in a neutralizer in 2,5 hours. In this case, as for changed washing and a washing process after putting in a neutralizer in 3.5 hours. In this case, as for case, the particle diameter of 17 ppm and sodium salt of the sodium element content in the obtained except having changed washing and a washing process after putting in a neutralizer in 1 hour. In this a neutralizer were changed in 1.5 hours, and it carried out like Example 1 except it. In this case, as the sodium element content in the obtained interlayer, the particle diameter of 0.5 micrometer and [0150]In preparation of resin of example 3 Example 1, it carried out like Example 1 except having [0151]In preparation of resin of example 4 Example 1, it carried out like Example 1 except having potassium sait of the particle diameter of 13 ppm and sodium sait was less than 0,5 micrometer, the sodium element content in the abtained interlayer, the particle diameter of less than 0.5 and potassium salt of the particle diameter of 0.9 ppm and sodium salt was less than 0.5 micrometer. potassium salt of the particle diameter of 3 ppm and sodium salt was less than 0.5 micrometer. [0152]In preparation of resin of comparative example 1 Example 1, it carried out like Example 1 interlayer was 8 micrometers.

was 13 micrometers. The evaluation result of the above Examples 1-4 and the comparative examples particle diemeter of 35 ppm and sodium salt of the sodium element content in the obtained interlayer except having changad washing and a washing process after putting in a neutralizer in 0.5 hour. The [0153]In preparation of resin of comparative example 2 Example 1, it carried out like Example 1 and 2 was shown in Table 2.

0.154

[Table 2]

			5	10 m		E. 92.86	E
		em)	57	20	w	para,	
<u>. </u>	デナターシ級定義数	•	~	***	Ų,	20,	701
ĕ	CK184 (388) 38 24 4 . 5	65.2	68.3	6,39	67. B	65, 3	85, 0
ŧ	平均第台家	S.	1336	1238	923	3,733	3700
ē	ゼニルアセチート液が (200.1%)	!. i	7	1.1	~	<u></u>	7
E	可多数是 (近後在)	2	Ç	48	8	â	ŝ
1	ナトリウム機関(ppb)	30	22	ø	3,3	13	ယ္သ
55	チトリウム教務(AD)		ю	so æ	8, 53	io.	22
	カリラム教徒(9920)	3,5	i i	9,5	3, 5	į	ŧ
	カリケム粒径 (44)	0.62	6, 35	Ĝ ≈	3.57	Î	ŧ
€	合わせがかる くペンメル級)	œ	æ	æ	ဘ	egs.	æ
粢	大阪落のよる際ののヘイズ(多)	85 85	83	Z.	, * ?	£ .	33
×	然合程度	0	ပ	٥	0	×	×

[0155]The maisture resistance excellent in Examples 1–4 is shown so that clearly from the above mentioned result. 10156 jedding the average degree of polymerization 1700, and the degree's of polyvinyl sloohol 275g of th dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chlorida saponification of 98.9 mol to the preparation pure water 2890g of Example 5 (1) resin --- warming --catalysts [201 g of] and the n-butylaidehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction

gree of acetalization) of this resin was 65.0-mol %, and the vinyl acetate component was 1.1-mol ylakdehyde is flushad. The potessium hydroxide solution which is a general-purpose neutralizer plete, the water of an excess (it is 30 times the amount to resin) washes, and unreacted ntrailzed the chloride catalyst, and also white powdered polyvinyl butyral resin was obtained The vinyl-acetal ingredient ough rinsing and desicoation for 2 hours with superfluous water.

ried out for 30 minutes, and 150 ** of 0.76-mm-thick interlayers were obtained. The bottom was a ying out melt kneading of this with a roll mill, with the press-forming machine, press forming was 57%2) Manufacture above-mentioned polyvinyl-butyral-resin 160 weight section of an interlayer, nental content of this interlayer. The particle diameter of less than [0.5 micrometer] mum and ar mixing triethylene-glycol-di-2-ethyl-butyrata 40 weight section as a plasticizer and fully. oft and 23 ppm in fixed quantity in ICP luminescence ultimate analysis about the potassium assium salt of the particle diameter of the sodium salt in an interlayer was less than 3

utes at 80 more **. Thus, this adhesion was performed for the glass laminate by which preliminary esion was carried out for 20 minutes on pressure 12 kg/cm² and conditions with a temperature of minutes, it moved to 90 4* oven, deaerated, and the vacuum press was carried out, halding for 30 inste with a transparent float gless (30 cm by 30 cmx25 mm in thickness), After putting this in in rubber bag and carrying out indirect desulturization mind with the degree of vacuum of 20torr for 58X3) It inserts from both sides using the manufacture above-mentioned interlayer of a glass ** in air type autoclave, and the transparent glass laminate was obtained

potassium elemental content in the obtained interlayer, the particle diameter of 1 micrometer and ium salt of the particle diameter of 5 ppm and potassium salt was less than 0.5 micrometer. nged washing and a washing process after putting in a neutralizer in 3.5 hours. In this case, as for ngod washing and a washing process after putting in a neutralizer in 2.5 hours. In this case, as for rometer and sodium salt of the particle diameter of 0.7 ppm and potassium salt was less than 0.5 593in preparation of resin of example 6 Example 5, it carried out like Example 5 except having 60]in preparation of resin of example 7 Example 5, it carried out like Example 5 except having potassium elemental content in the obtained interlayer, the particle diameter of less than 0.5

opt having changed washing and a washing process after putting in a neutralizer in 1 hour. In this e, the particle diameter of 104 ppm and potassium salt of the potassium elemental content in the 613n preparation of resin of comparative example 3 Example 5, it cerried out like Example 5 ained interlayer was 6 micrometers.

apt having changed washing and a washing process after putting in a neutralizer in 0.5 hour. In this the particle diameter of 220 ppm and potassium salt of the potassium elemental content in the \$2]in preparation of resin of comparative example 4 Example 5, it carried out like Example 5 ained interlayer was 9 miorometers.

63]About the place laminate obtained by the above Examples 5~8 and comparative examples 3 and n adhesive examination (pan Meru examination) and a humidity test are done by the same method

the above-mentioned, and the evaluation result is summarized in Table 3, and is shown

64] bie 3]

3//www.f.ipdi.npit.go.jp/ogi-bin/tran_web_cgi_ejje?atw_u=http%3A%2F%2FwwwA.jpdi... 2009/06/15

JP.11-343152,A [DETAILED DESCRIPTION]

18/40 ページ

-	***************************************					
		24,	发烙纸		元教堂	<u>%</u>
		in.	φ	j.	23	Arie.
	アセラール基炭素数	*	v	~	*	~
	アセタール作成(80)公	ဇ (ရှိ	0 (9	85.8	85. B	6.53
æ	平均查合度	1700	1366	1706	17.05	3773
200	ビニルアセテート概念 (mo 1%)	<u>ئ</u> ت	~; 	 	1,1	2
ž	可经常是 (成品化)	\$	ş	\$	ş	*
%	カリウム遊(度(ppm)	83	.05	6.3	20	223
	カリウム報経 (10の)	270	-	\$2 °6	:ca	*
	ナトロウム機関(sps)	 .::	8. 9.	8,5	;	-3
	ナトリウム教強(gan)	8.8	ુ ક	\$ 20	1	1
4 2	合わせガラスの接着性 (パンノル値)	00	æ	æ	∞.	
18	水液後2.4時間後のヘイズ (%)	ä	92	Ξ	æ	26
*	移合构定	0	0	0	×	×

[0165] The moisture resistance excellent in Examples 5—7 is shown so that clearly from the above—

complete, superflusus water washes, and unreacted n-butylaldehyde is flushed. The sodium hydroxide [0166]adding the average degree of polymerization 1780, and the degree% of polyvinyl aloohol 2.75g of powdered polyvinyl butyral resin was obtained through rinsing and desicostion with superfluous water. it dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chlorida catalysts [201 g of] and the n-butylaldehyde 148g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction solution which is a ganaral-purpose neutralizer neutralized the chloride catalyst, and also white saponification of 98.9 mol to the preparation pure water 2890g of Example 8 (1) resin The degree of acetalization of this resin was 65.0-mol %.

forming was cerried out with the press-forming machine, and the interlayer was obtained. The bottom above-mentioned polyvinyl-butyral-resin 100 weight section of the interlayer as a plasticizer, press was a result and 13 ppm in fixed quantity in the IOP luminescence elemental analysis davice about [0167](2) Triethylene-glycof-di-2-ethyl-butyrate 40 weight section was mixed with manufacture the sodium element content of this interlayer. The particle diameter of the sodium salt in an interlayer was 3 micrometers,

[0168](3), pinching the manufacture above-mentioned interlayer of a glass laminate between the 2.5narformed for 20 minutes on condition of the temperature of 60 **, and pressure " of 5kg/om, and deaeration under decompression of ~600mmHg in autoclave. Heat arimping was simuitaneously mm-thick float glasses of two sheets, putting this in in a rubber bag, and carrying out suction the giass laminate was produced.

like Example 9 except the temperature at the time of heat orimping having been 80 **. The particle [0169] is production of the glass laminate of example 9 Example 8, the glass saminate was produced diameter of the sodium sait in an interlayer was 3 micrometers.

[0170] in production of the glass laminate of example 10 Example 8, the glass laminate was produced like Example 8 except the temperature at the time of heat crimping having been 100 **. The particle diameter of the sodium sait in an interlayer was 3 micrometers.

[0171] in preparation of reak of comparative example 5 Example 8, except having changed washing was produced. In this case, the particle diameter of 30 ppm and sodium selt of the sodium element and a washing process for a short time a little, it carried out like Example 8 and the glass laminate content in the obtained interlayer was 11 micrometers.

72]About the glass laminate obtained by the above Examples 8-10 and comparative example 5, adhesive examination (pan Meru examination) and the heat resistance test were done by the wing methods. About the humidity test, it carried out like Example 1.

inate is uniformly hit by a hammer head, the area of the glass piece adhering to the interlayer of a 73]After cooling a valuation method (1) edhesive-moperty (pan Meru) examination glass laminate tom is carried out with the curvature which is 50 mm in radius, the used automatic hammer ring is laminate is chaerved visually, and adhesive strength (pan Meru value) is judged as contrasted 20 ** for 2 hours, An automatic hammer ring device is equipped, the whole surface of a glass ice has a hammer head with a blow effective diameter of 5 mm and a weight of 240g, and the dimum of a grade sample is a ten-point method of ten points. Curved-surface shaping of the I the grade sample shown in Table 1. The adjustive strength minimum is one point and the king power of a hammer head oan be adjusted with a spring screw.

inate." The result of the above Examples 8-10 and the comparative example 5 was summerized in 74](2) Take out end observe the existence of foaming visually, after neglecting the obtained glass hate under 130 ** atmosphere for 2 hours based on the heat resistance test. IIS R3205 "glass

de 4, and was shown.

	जन	光施宏		;; \$	
	80	*	ت 	u>	
トリウム微度(ppg)	22	23	2	35	~~~
トリウム粒谷(410)	20	20	670	=	
わせガラスの被奪性 (パンメル協)		7C	540	uti	
発像2.4時間後のヘイズ (%)	ĸ	3%	22	88	
らせガラスの凝熱性試験 の発泡の有解	.) #8	.:) 429	3	بة ت	
合判定	ာ	0	0	×	

yvinyl butyral resin was obtained for this resin through the desicoation under the atmosphere of 60 16]adding the everage degree of polymerization 1769, and the degree's of polyvinyl alcohol 275g of onification of 98.9 mol to the preparation pure water 2894g of Example 11 (1) resin --- warming --issolved Temperature control of the system of reaction was carried out to 12 **, 35% of chloride thed with the centering dehydrator and resin of water content was obtained 50%. White powdered alysts [201 g of] and the n-butylaidehyde 148g were added, this temperature was held, and the ylaldehyde was flushed. The ph value was 5.1 when ph of the system at this time was measured. de to complete, the water of the excess (they are 30 times to resen) washed, and unreacted nctant was deposited. Then, held the system of reaction at 45 ** for 3 hours, the reaction was and ~700mmHg. The degree of acetalization of this resin was 65.0-mol %.

sodium element content in this interlayer. The particle diameter of the sodium saft in an interlayer ming was carried out with the press-forming machine, and the interlayer was obtained. The bottom overmentioned polyvinyl-butyral-resin 180 weight section of the interlayer as a plasticizer, press s a result and 0.7 ppm in fixed quantity in the ICP luminescence elemental analysis device about 77)(2) Triethylene-glycol-di-2-ethyl-butyrate 40 weight section was mixed with manufacture s less than 0.5 micrometer.

78)(3), pinching the manufacture above-mentioned interlayer of a glass faminate botween the 2,5ieration under decompression of ~600mmHg in autoclave. Heat crimping was simultaneously r-thick float glasses of two sheets, putting this in in a rubber bag, and carrying out suction

19]adding the average degree of polymerization 1700, and the degrees of polyvinyl alcohol 275g of formed for 20 minutes on condition of the temperature of 60 **, and pressure 2 of 5kg/cm, and glass laminate was produced.

p://www4.indlinpit.go.jn/ogr-bin/tran_web_rgi_ejjz?atm_u=http%3A%2F%zFwww4.ipG81... 2009/08/15

JP.11-343152.A [DETAILED DESCRIPTION]

reactent was deposited. Then, hold the system of reaction at 45 4* for 3 hours, and the reaction was ** of an excess (they are 30 times to resin), and white powdered polyvaryl butyral resin was obtained it dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chioride saponification of 98.9 mol to the preparation pure water 2890g of Example 12 (1) resin --- warming -catalysts { 201 g of] and the n-butylaldehyde 148g were added, this temperature was held, and the solution which is a general-purpose neutralizer. Then, it rinsed with water with a temperature of 50 butylaldehyde was flushed, and the chleride catalyst was neutralized using the sodium hydroxide made to complete, the water of the excess (they are 30 times to resin) washed, unreacted nthrough desiccation. The degree of acetalization of this resin was 65.0-mol %.

the sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer forming was carried out with the press-forming machine, and the interlayer was obtained. The bottom above-mentioned polyvinyl-butyral-resin 100 weight section of the interlayer as a plasticizer, press was a result and 10 ppm in fixed quantity in the ICP luminescence elemental analysis device about [0180](2) Triethylene-glycof-di-2-ethyl-butyrate 40 weight section was mixed with manufacture was 3 micrometers.

0181%3, pinching the manufacture above-mentioned interlayer of a glass laminate between the $2.5^$ performed for 20 minutes on condition of the temperature of 60 **, and pressure 5 kg/am², and the deaeration under decompression of -600mmHg in autoclave. Heat crimping was simultaneously mm-thick float glasses of two sheets, putting this in in a rubber bag, and carrying out suction glass laminate was produced.

used water with a temperature of 60 ** by washing after neutralization. The degree of acetalization of this interlayer was 650-mol %. The bottom was a result and 15 ppm in fixed quantity in the ICP [0182]In example 13 Example 12, the interlayer was obtained like Example 12 except [all] having luminescence elemental analysis device about the sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer was 4 micrometers.

the sodium element content in this interlayer. The particle diameter of the sodium salt in an interlayer [0183]adding the average degree of polymerization 1700, and the degrees of polyvinyl alcohol 275g of complete, superfluous water washes, and urreacted n-butylaldeliyde is flushed. Magnesium lydroxide forming was carried but with the press-forming machine, and the interlayer was obtained. The bottom it dissolved. Temperature control of the system of reaction was carried out to 12 **, 35% of chloride catalysts { 201 g of } and the n-butylaidenyde 148g were added, this temperature was held, and the above-mentioned polyvinyi-butyral-resin 100 weight section of the interlayer as a plasticizer, press was a result and 0.7 ppm in fixed quantity in the IOP luminescence elemental analysis device about. sapanification of 98.9 mol to the preparation pure water 2890g of Example 14 (1) resin --- warming reactant was deposited. Then, held the system of reaction at 45 ** for 3 hours, make a reaction [0184](2) Triethylene-glycol-di-2-ethyl-butyrate 40 weight section was mixed with manufacture superfluous water, and white powdered polyvinyl butyral resin was obtained through desiccation. which is a general-purpose neutralizer neutralized the chloride catelyst, and elso it rinsed with was less than 0.5 micrometer.

(0.185)(3), pinching the manufacture above-mentioned interlayer of a glass laminate between the $2.5^$ performed for 20 minutes on condition of the temperature of 60 **, and pressure 2 of 5kg/sm, and deseration under decompression of "600mmHg in autoclave. Heat crimping was simultaneously mm-thick float glasses of two sheets, putting this in in a rubber bag, and carrying out suction the glass laminate was produced.

[0186]About the glass laminate obtained in above Examples 11-14, the heat resistance test was done by the following methods. About the humidity test, it carried out like Example 1. However, Example 14 carried out only the humidity test.

heated in a 150 ** oil bath for 60 minutes, and the existence of degradation of resin was observed. [0187]The resin 1g was put into the valuation method (1) heat resistance test usual test tube, it The result of the above Examples 11-14 was summarized in Table 5, and was shown.

Table 5

http://www4.ipdlinpit.go.jp/ogi-bin/tran_web_ogi_eije?atw_u=http%3A%2F%ZFwww4.ipdli... 2008/06/15

11 12 13 14 トリウム松佐(pps) Q.T 10 15 0.7 トリウム松佐(np) B.S) 3 4 Q.S) 株性紅砂 内盤 NB			来游戏	\$2.	
88 88 88 88 9 9 1 1 1 1 1 1 1 1 1 1 1 1		~	512 144	65 444	-10°
8.50 C C C C C C C C C C C C C C C C C C C	トリウム器(近(1982)	9,7	c;	35	က် ကိ
20 20 20 C 20 C 20 C 20 C 20 C 20 C 20	トリウム粒径 (42)	8,55		v	6.5
8 0 8 0	秦 在京教 《西京教	設なる	第2 2 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3 3	親な	ł
0	侵費2.4時間後のヘイズ (光)		38	Œ	×
	奇物定	ं		0	ş

ABjadding the polyvinyl eloohol 275g of the average degree of polymerization 1700 and 0.1% of the ight of the degree % and sodium acetate content of saponification of 98.9 mol to the preparation e water 2890g of Example 15 (1) resin — warming — it dissolved. Temperature control of the dem of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the n-yaldehyde 140g were added, this temperature was held, and the reactant was deposited. Then, if the system of reaction at 45 ** for 3 hours, make a reaction complete, superfluous water (they 30 times to resin) washes, and unreacted n-butylaldehyde is flushed, After neutralizing a chloride salvst using sodium hydroxide which is a general-purpose neutralizer, superfluous water washed is white powdered polyvinyl butyral resin was obtained through desicoation. The degree of talization of this resin was 85-mol %.

90%2) Triethylena-giyeal-di-2-ethyl-butyrate 40 weight section was mixed with manufacture overmentioned polyvinyl-butyral-resin 100 weight section of the interlayer as a plasticizer, press ming was carried out with the press-forming machine, and the interlayer was obtained. The bottom is a result and 8 ppm in fixed quantity in the ICP tuminescence elemental analysis devise about the flum element content in this interlayer.

91]The interlayer was obtained like Example 15 except having used the polyvinyl alcohol 275g of example 16 avarage degree of polymerization 1700 and 0.4% of the weight of the degree % and lium exetate unitent of seponification of 98.9 mol. The degree of acetalization of this interlayer s 65.0-mol %. The bottom was a result and 13 ppm in fixed quantity in the ICP juminescence mental analysis device about the sodium element content in this interlayer.
92]adding the polyvinyl alcohol 275g of the average degree of polymerization 1709 and 0.1% of the gift of the degree & and sodium acetate content of sample 498.9 mol to the preparation was a sequence.

e water 2890g of Example 17 (1) resin — warming — it dissolved. Temperature control of the ten of reaction was carried out to 12 **, 35% of chloride catalysts [201 g of] and the n-ylabshyde [48g were added, this temperature was held, and the reactant was deposited. Then, of the system of reaction at 45 ** for 3 hours, make a reaction complete, superfluous water (they 30 times to resin) washes, and unreacted n-butylaidehyde is flushed, After neutralizing a chloride adject using octanoic acid magnesium which is a neutralizer, superfluous water washed and white videred polyvinyl butyral resin was obtained through desiccation. The degree of acetalization of this in was 55-mul %.

93(2) Triethylene-gyoof-di-2-ethyl-butyrate 40 weight section was mixed with manufacture re-mentioned polyvinyl-butyral-resin 100 weight section of the interlayer as a pissticizer, press ming was carried out with the press-forming machine, and the interlayer was obtained. The bottom is a result and 2 ppm in fixed quantity in the ICP luminescence elemental analysis device about the flum element content in this interlayer.

94]The interlayer was obtained like Example 17 except having used ethyleneoxide as a nautralizer in example 18 chloride catalyst. The degree of socialization of this interlayer was 65-mol %. The tom was a result and 2 ppm in fixed quantity in the IOP luminescence elemental analysis device sit the sodium element content in this interlayer. Using the interlayer obtained in above-mentioned unples 16-18, the same heat resistance test as Example 11 and the same humidity test as imple 1 were done, and the result was shown in Table 6.

. G 2://www4.ipdl.npit.go.jp/cgi-bin/tran_web_cgi_eije?atm_u-http%34%2F%3Fwww4.ipdl.... 2008/06/15

JP.11~343152,A [DETAILED DESORIPTION]

22/40 ページ

の :			
の の の の の の の の の の の の の の	•	E-	80 ~√
8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8		 ej	£.
ж XX б XX		-82	φá
30.	ę.	: ::	3.5
	認な。 超し	観点	競び
水浸液2.1年間後のヘイズ 19 35 (※)	32 23		: <u>e</u>
() () () () () () () () () ()	<u>்</u>	Q	0

[0196]adding the average degree of polymerization 1700, and the seponffication degree% of polyvinyl elochol 275g of 98.9 mol to the preparation pure water 2890g of example 19(1) polyvinyl-acetal resin—warming—it dissolved. Temperature pentral of the system of reaction was carried out to 12 **, 35% of the weight of chloride catalysts [20f g of] and the n-butyladdehyde 146g were added, this temperature was held, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, make a reaction complete, superfluous water washes, and unreacted n-butylaidehyde is flushed. The sodium hydroxide solution which is a general-purpose neutralized n-butylaidehyde is catalyst, and white powdered polyvinyl butyral resin was obtained through rinsing and desiscation for 2 hours with still mare superfluous water. The smount of 64-mol % and survival scetyl groups of the

degree of formation of average butyral of this resin was 1-mol %.

[0197](2) the polyvinyl butyral resin (average-degree-of-polymerization 1700 and degree % of 64 mol of formation of average butyral, obtained by the maintfacture above of the interlayer for glass laminates 0.86 copy of octanoic acid magnesium is added as a plasticizer to amount of residual acetyl groups 1 mol %100 copy as 40 copies of triethylene glycol gir-ethyl butyrate, and an adhesive regulator. After fully carrying out melt knesding with a roll milt press forming was parried out for 30 minutes at 150 ** using the press-forming machine, and the interlayer for glass laminates of 0.76 mm of average thickness was obtained. The particle diameter of 10 gpm and sodium salt of the sodium content in the obtained interlayer for glass laminates was 1 micrometer. It was 0.9 micrometer when the particle diameter of the magnesium salt which exists in an interlayer was measured using the time-of-flight type secondary-ion-mass-spectroscopy device (TOF-SMS).

(0198)(3) The interlayer for glass laminates obteined by the manufacture above of the glass laminate, After having put from the both sides with the transparent float glass (30 cm by 30 cmx3 mm in thickness), putting this in the rubber hag and carrying out indirect desuffurization mind with the degree of vacuum of 20torr for 20 minutes, it moved to oven, descrated, and the vacuum press was carried out, holding for 30 minutes at 90 more **. Thus, this sticking by pressure was performed for the glass laminate by which preparative pressure arrival was carried out for 20 minutes on condition of the temperature of 135 **, and pressure 12 kg/om² in air type sutoclave, and the glass laminate was obtained.

101931in manufacture of the interlayer for example 20 glass leminates, the interlayer for glass laminates and the glass laminate were obtained like Example 19 instead of 0.08 copy of octanoic acid magnesium as an adhesive regulator except having added 0.09 copy of neo decanoic acid magnesium. It was 0.5 micrometer when the particle diameter of the magnesium saft which exists in the interlayer for glass laminates obtained above was measured like Example 19.

[0200]In manufacture of the interlayer for example 21 glass laminates, the interlayer for glass laminates and the glass laminate were obtained like Example 18 instead of 0.08 copy of octanoic acid magnesium as an adhesive regulator except having added 0.04 copy of magnesium chloride. It was 2 micrometers when the particle diameter of the magnesium saft which exists in the interlayer for glass laminates obtained above was measured like Example 19.

9201]In manufacture of example 22 polyvinyl–acetal resin, as a neutralizer, Instead of sodium bydroxide solution, the everage degree of polymerization 1700, and% of the powdored amount white

http://wwwf.ipdl.inpit.go.jp/ogi-bin/tran_web_ogi_ejje?atw_u=http%34%2F%2Fwwwf.ipdl.i.. 2009/06/15

ained like Example 19 using 100 copies of polyvinyl butyral resin obtained above except not having led 0.08 copy of octanoic acid magnesium as an adhesive strength regulator. The particle diameter 3.7 ppm end sedium salt of the sodium content in an interlayer was less than 0.5 micrometer. It. nsequently, instead of 100 copies of polyviny! butyral resin obtained in Example 19 in manufacture the interlayer for glass laminates. The interlayer for glass laminates and the glass faminate were vviry! butyral resin of degree % and survival acety! groups of formation of average butyral of 64 s 2 micrometers when the particle dameter of the magnesium saft which exists in an interlayer of 1 mal were obtained like Example 19 except having used besic magnesium carbonate. s measured like Example 19.

ained like Example 19 using 100 capies of polyvinyl butyraf resin obtained above except not having led 0.08 copy of actanolo acid magnesism as an adhesive strength regulator. The particle diameter osequently, instead of 100 copies of polyvinyl butyral resin obtained in Example 19 in manufacture troxide solution, the average degree of polymerization 1700, and% of the powdered amount white he interlayer for glass laminates. The interlayer for glass laminates and the glass laminate were yvinyl butyral resin of degree % and survival acetyl groups of formation of average butyral of 64 of 1 mol were obtained like Example 19 except having used the magnesium hydroxide solution. promoters when the particle diameter of the magnesium salt which exists in an interlayer was 02]In manufacture of example 23 polyvinyl-acetal resin, as a neutralizer, instead of sodium 1.7 ppm and sodium salt of the sadium content was less than 0.5 micrometer. It was 2.5 asured like Example 19,

particle diameter of the magnesium saft which exists in the interlayer for glass laminates obtained in or a plasticizer instead of 0.08 copy of octanole soid magnesium. It was 10 micrometers when 03]In manufacture of the interlayer for comparative example 6 glass faminates, as an adhesive ulator. The interlayer for glass laminates and the glass laminate were obtained like Exemple 19 ept having added 0.04 copy of magnesium acetate which is hard to dissolve in polyvinyl butyral we was measured like Example 19.

in system organio ecid which are hard to dissokve in polyvinył butyrel resin or a plesticizer instead opt having added 0,05 copy of butancic acid which is 0,04 copy of magnesium acetate and short gnesium salt which exists in the interlayer for glass laminates obtained above was measured like 04) in manufacture of the interlayer for comparative example 7 glass faminates, as an adheaive ulator. The interlayer for glass laminates and the glass laminate were obtained like Example 19 3.08 copy of octanolo acid magnesium. It was 4 micrometers when the particle diameter of the

aparative examples 6 and 7 was done like Example 1. The result was as being shown in Table 7. 05] The humidity test of the interlayer for glass faminates obtained by Examples 19-23 and the 8

		30.	% %			ž	200
	4	0.2	~	54 22	25 24	ග	ţ»
アセクール基依案数		**	*	*	*	۰	7
年均ブチラール位置 (モル第)	6 X	64,0	e Z	÷	0 7	26.00	3,4
気体アセチル構造 (そんな)	9	۵	۵ ۲	8	ex	9 %	83 ~
學物質会強	1709	. 78	202	9021	150	3708	3238
可缩微器 (数量张)	2	3	ş	ş	\$	₽	æ
チャリケム器器(sps)	ä	£	Ξ	 ed	بر و	٤	Œ
ナトリウム統領 (325)		να •~	ند	ф Э	3	3	1.0
マゲネンウム塩液加度 (電解的)	80 %	83 65	్రా త	804	æ.	25.00	స జ
マグネンウム版の総径 (元的)	gu er	9,6	\$ 5	હ્ય છે	ક સ	36.8	3
乗2.4 時間後のヘイズ (%)	58.8	18,4	26.8 8	88 88	38.5	88, 80,	37.2
#\Z	٥	o	٥	a	a	×	×

1//www4.ipdl.inpit.go.jp/cgi-bin/tran_web_cgi-ejje?atw_u=http%3A%2F%2Fwww4.indli... 2009/06/15

JP,11-343152,A [DETAILED DESORIPTION]

24/40 4-5

examples 8 and 7 with the particle diameter of greater than 3 micrometers of the magnesium salt esistance so that clearly from Table 7. On the other hand, the glass laminate of the comparative [0207] The glass laminate of Examples 19-23 by this invention showed the outstanding moistura which exists in a glass laminate interlayer had bad moisture resistance

alcohol of seponification of 99.2 mol 198 weight section (4.5 mol of vinyl alcohol considerable amount) 1706. After cooled this solution at 30 **, having added 35-% af the weight chloride 208 weight section were performed, and the degrees of polyvinyl butyral resin of butyral-izing of 65 mol was obtained. It subsequently to 2 **, holding this temperature and polyviny! butyral resin's depositing, temperature (0208]Example 24 composition / preparation (composition of polyvinyl butyral resin) ion-exchangeup of the solution temperature was carried out to 30 **, and it was held for 5 hours. After holding, was 50 ppm when the sodium content of the obtained polyviny! butyral risin was measured by 10P sodium bicarbonate 158 weight section (1.8 mol) was added, it neutralized, rinsing and desiccation to the resector with an agitating devine, and agitating it with the average degree of polymerization water 2900 weight section, it heated and dissolved in 95 **, having supplied degrees of polyvinyl (2.1 mol) and n-butylaideliyde 152 weight section (2.1 mol), lowering solution temperature emission spectrometry. The particle diameter of sodium salt was 12 micrometers,

[0209](Production of a resin layer) Polyvinyl-butyral-resin 100 obtained weight section, Tricthylenecarried but for 30 minutes on condition of 150 ** and 120 kg/om² with the press-forming machine, Press forming of the kneeded material produced by supplying 2-ethylbutanoic acid magnesium 0.04 gycol-di-Z-ethyl-butyrate 40 weight section, ethylenediaminetetraecetic acid 0.05 weight section, and the 0.9-mm-thick resin layer was obtained. About the obtained resin layer, the same humidity test as Example 1 was done. As modified silicone oil, what is expressed with a following chemical weight saction and modified silicone oil 0.05 weight section to a roll mill, and kneading them was formula was used.

[Formula 1]
$$CH_{1} = \begin{cases} CH_{1} & CH_{2} \\ CH_{3} = O + \frac{C}{4} = O + \frac{C}{4} = O + \frac{C}{4} = CH_{3} \\ CH_{3} & CH_{3} = O + \frac{C}{4} = O + \frac{C}{4} = CH_{3} \end{cases}$$

(0211)Instead of example 25 ethylenediaminetetrascetic-acid 0.05 weight section, the resin layer was obtained and evaluated like Example 24 except having used salicylaidehyde 0.08 weight section. The result was shown in Table 8.

10212]Instead of example 26 ethylenediaminetetrascetic-acid 0.05 weight section, the resin layer was obtained and evaluated like Example 24 except having used oxalic acid 1.0 weight section. The result was shown in Table B.

[0213]Instead of example 27 ethylenediaminetetrascetic-acid 0.05 weight section, the resin layer was obtained and evaluated like Example 24 except having used 1,10-phenanthroline 0.03 weight section. The result was shown in Table 8.

[0214]instead of example 29 ethylenediaminetetrascetic-acid 0.05 weight section, the resin layer was obtained and evaluated like Example 24 except having used acetylacetone 0.3 weight section. The result was shown in Table B.

comparative example 8 ethylenediaminetetrascetic-acid 0.05 weight section. The result was shown in [0215]The resin layer was obtained and evaluated like Example 24 except not having used Table 8.

10216 Unstead of comparative example 9 ethylenediaminetetranectic acid 0.05 weight section, the resin layer was obtained and evaluated like Example 24 except heying used acetone 0.1 weight section. The result was shown in Table 8.

able 8

http://www.hipdl.inpit.go.jp/cgi-bin/tran.web.cgi-ejje?atw.u=http83JA82F%2Fwww4.io@l... 2009/06/15

大次後の人の発記	(S)	28.0	2.8	35, 6	3.00	98.0	77.0	# 5. #
なべる。	が金銭の金銭(総数)	4, 65	0, 65	0. 0.	9 ° 8	કું ઉદ	0, 65	රා ක න
2467880 2475880	(高麗麗)	9 . C	9. S. 4	** &	9, 0, 8	8 8 8	I	ì
	多金彩	18 19 18 18	8 8 3	6 1	e 0 0	0, 3	i	 200
希体形成化合物	機器	442771、7回路路集	*5\$\$7\$5\$	ショの酸	E, 18-72508897	アセチルアセトン 0.3	- - - -	7.48%
ジャステル		9	3	0 *	8	4.8	8 +	4.0
ブチラール	(金銭)	198	100	100	1.0 8	क स -	138	138
		* %	ec.	တ္ မ	23 12-	æ ∾	'nО	G

18]adding the average degree of polymerization 1700, and the degree% of polyvinyl alcohol 275g of onfification of 98.9 mot to the preparation gure water 2890g of example 29(1) polyvinyl-acetal resin warming —— it dissolved. Temperature control of the system of reaction was carried out to 12 **s., if g of 35-% of the weight chloride catalysts and the rr-butylaidehyde 148g were added, this operature was held, and the reactant was deposited. With superfluous water, field the system of ction at 45 ** for 3 hours, make a reaction complete, wash, and Then unreacted in-butylaidehyde shout, The sodium hydroxide solution which is a general-purpose neutralizer neutralized the oride catalyst, and also white powdered polyvinyl butyral resin was obtained through missing end degree of formation of average butyral of this resin was 1-mol % and survival scettyl groups of degree of formation of average butyral of this resin was 1-mol %.

19](2) manufauture profit **** polyving butyral resin (average-degree-of-polymerization 1700) I degree % of 64 mol of formation of average butyral, of the interlayer for glass laminates. To court of residual acetyl groups 1 mol %100 copy, as a plasticizer 40 copies of triethylene glycol direktyl butyrate, And 0.13 copy of dimethyl octylamine is added as 0.75 copy of lecyllenzenesulfamic soid, and amilia as organic soid, After carrying out melt kneeding enough with all milk press forming was carried out for 30 minutes at 150 ** using the press-forming machine, the interlayer for glass laminates of 0.78 mm of average thickness was obtained. It was 80 ppm

If the interlegive for glass laminates a begants and surprise out the measures around and, and animates at 150 ** using the press-forming machine, if the interlegive for glass laminates of 0.76 ** using the press-forming machine, if the interlegive for glass laminates of 0.76 ** using the press-forming machine, and the sodium content of the obtained interlayer was measured by ICP emission spectrometry. It is 4 micrometers when the particle diameter of the sodium element which exists in an interlayer is measured using the time-of-flight type secondary-ion-mass-spectroscopy device (TOF-SIMS) 201(3) it inserts from both sides using the manufacture above-mentioned interlayer of a glass instead with a transparent float glass (30 cm by 30 cmx3 mm in thickness). After putting this in similates bag and carrying out indirect desulfurzation mind with the degree of vacuum of 20ther for minutes, it moved to 90 ** even, deserated, and the vacuum press was carried out, holding for 30 utes at 80 more ** Thus, this adhesion was performed for the glass laminate by which preliminary nesion was carried out for 20 minutes on condition of the temperature of (35 **, and pressure 12).

cm² in air type autoclave, and the glass laminate was obtained. About the obtained glass laminate valayer, the humidity test was done like Example 1 and the result was shown in Table 9.

21]In manufacture of the interlayer for example 30 glass laminates, 0.30 copy of octanels acid is sed instead of 0.75 copy of dodecythenzenesultonic acid as organic acid. Except having added 0.35 w of decyl amine instead of 0.13 copy of dimethyl octylamine as amine, the resin layer was aired like Example 29 and the evaluated result was shown in Table 9. It was 5 micrometers when particle diameter of the sodium element which exists in the obtained interlayer was measured like 129.

2.2.In manufacture of the interlayer for example 31 glass laminates, except having added 0.20 oxpy 38.2-ethylhexyl) phosphoric acid instead of 0.15 copy of dodscyßenzenesulfonin acid as organic 4, the resin layer was obtained like Example 29 and the evaluated result was zhown in Table 9. It is 2 micrometers when the particle diameter of the sodium element which exists in the obtained arlayer was measured like Example 29.

23]In manufacture of the interlayer for comparative example 10 glass laminates, except not having to the organic ecid and amine of the dispersing agent, the resin layer was obtained like Example and the evaluated result was shown in Table 9. It was 20 micrometers when the particle diameter

0.//wwwf.ipdl.inoit.go.jo/ogi-bin/tran_web_cgi_ejje?atw_u=http%3A%2F%2Fw2Fww4.ipdl... 2009/08/15

of the sadium element which exists in the obtained interlayer was measured like Example 29. [0224] in manufacture of the interlayer for comparetive example 11 glass laminates, except not having added the organic soid and emine of the dispersing agent, and having changed washing time from 2 hours, in 3 hours, the resin layer was obtained like Example 29 and the evaluated result was shown in Table 9. It was 30 ppm when the sodium content which exists in the obtained interlayer was measured like Example 29. It was 13 micrometers when the particle diameter of the sodium element was massured like Example 29.

102203 (Table 93

		रणः	光物気		20	比較例
		00 04	න ග	~~ 671	æ 	~
	アセタール基板系数	-*	~*	***	~2	*:
	学数プチラール化数 (4.8%)	6) (6)	2 Ye	84.0	6.1.0	6.
45	模存でセテル基盤 (モルX)	0		8	 	:es
9	學均強合度	3706	3700 3708	1735	230	1700
æ	可發素質(海路器)	ą,	1	Ş	Ž,	*
200	ナトリウム後漢(sopu)	簽	98	33	æ	۶
	チトリウム散発(仏政)	*	(C)	e)	≋	23
X	水浸泡さり時間後のヘイズ	38	83	20	81	35
1	緣合裡定	ୁ	ာ	ာ	X	×

slochel of sepenification of 99.2 mel 198 weight section (4.5 mel of vinyl slechel considerable amount) 700. After cooled this solution at 30 %, having added 35-% of the weight chloride 196 weight section were performed, and the degrees of polyviny! butyral rosin of butyral-izing of 65 mol was obtained. It press-forming machine, and the 0.8-mm-thick resin layer was obtained. The obtained resin layer was [0227](Production of a resin layer) Palyvinyl-butyral-resin 1011 obtained weight section, Triethyleneglycol-di-2-zthyi-butyrate 40 weight section, p-toluonesulfonic-acid 0.43 weight section, And press subsequently to 2 **, holding this temperature and polyviny! butyral resin's depositing, temperature forming of the kneaded material produced by supplying hexylamina 0.23 weight section to a roll mill, :0226]Example 32 composition / preparation (composition of polyvinyl butyral resin) ion-exchangewas 50 ppm when the sodium content of the obtained polyviny butyral resin was measured by IOP up of the solution temperature was carried out to 30 **, and it was held for 5 hours. After helding, sodium bicarbonata 147 weight section (1,7 moi) was added, it neutralized, rinsing and desiccation used and the moisture-proof white-blush-mark sex test was done like Example 1. The result was water 2900 weight section, It heated and dissolved in 95 **, having supplied degrees of polyvinyl to the reactor with an agitating device, and agitating it with the everage degree of polymerization and kneading it was carried out for 10 minutes on condition of 150 ** and 120 kg/cm² with the (1.8 mal) and n-budylaldefiyde 152 weight section (2.1 mal), fowering solution temperature emission spectrometry. The particle diameter of sodium salt was 12 micrometers.

shown in Table 10. [0228]Instead of example 33 hexylamine 0.23 weight section, the resin layer was obtained and evaluated like Example 32 except having used tetradecylamine 0.49 weight section. The result was shown in Table 10.

[0229]Instead of example 34 p-toluenosulfonio-acid 0.43 weight section, the resin layer was obtained and evaluated like Example 32 except having used dodecylbenzenesulfanic acid 0.75 weight section. The result was shown in Table 10.

[0230]The resin layer was obtained and evaluated like Example 32 instead of example 35 problemesulfonic-acid 0.43 weight section end hexylamine 0.23 weight section except having used

lecybenzenesuifonin acid 0.75 weight section and decyl amine 0.36 weight section. The result was tenesulfonic-acid 0.42 weight section and hexylamine 0.23 weight section except having used 31] The resin layer was obtained and evaluated like Example 32 instead of example 38 p-

dacylbanzenesulfonic acid 0.75 weight section and dodecyl amina 0.42 weight section. The result renesationic—soid 0.43 weight section and hexylamine 0.23 weight section except having used 32]The resin layer was obtained and evaluated like Example 32 instead of example 37 pshown in Table 10.

ecybenzenesulfonio acid 0,75 weight section and N,N-diocty! amine 0.55 weight section. The enesulfonic-acid 0.43 weight section and hexylamine 0.23 weight section except having used 33)The resin layer was obtained and evaluated like Example 32 instead of example 38 puft was shown in Table 10.

34]Instead of example 39 p-toluenesulfonic-acid 0.43 weight section and hexylamine 0.29 weight lecythenzenesulfania acid 0.37 weight section and N.N.-dimethy! ontylamine 0.18 weight section. tion. The resin layer was obtained and evaluated like Example 32 except having used s result was shown in Table 10.

35]instead of example 40 p-toluenesulfonio-acid 0,43 weight section and hexylamine 0.23 weight. Aton, The resin layer was obtained and evaluated like Example 32 except having used ecybenzenesufonic acid 0.75 weight section and N.N-dimethyl octylamine 0.36 weight section. s result was shown in Table 10.

lecylbenzanesulfonic acid 0.75 weight section and N.N-dimethyldodecyl amine 0.49 weight section. 36]instead of example 41 p-toluenesulfanic-acid 8.43 weight section and hexylamine 0.23 weight thon. The resin layer was obtained and evaluated like Example 32 except having used s result was shown in Table 10.

nparative example 12 p-toluenesulfanic-acid 0.43 weight section and hexylamine 0.23 weight 37]The resin layer was obtained and evaluated like Example 32 except not having used tion. The result was shown in Table 10.

in layer was obtained and evaluated like Example 32 instead of hexylamine 0.23 weight section 383 Comparative example 13 p-towenesulfonic-acid 0.43 weight section was not used, but the 39)The resin layer was obtained and evaluated like Example 32 except not having used rept having used decyl amine 0.36 weight section. The result was shown in Table 10.

40]The resin layer was obtained and evaluated like Example 32 instead of comparative example 15 oluenesuffanic-acid 0.43 weight section and hexylamine 0.23 weight section except having used nparative example 14 hexylamine 0.23 weight section. The result was shown in Table 10.

41]The resin layer was obtained and evaluated like Example 32 instead of comparative example 16 oluenesulfanic-acid 0.43 weight section and hexylamine 0.23 weight section except having used lium dodecylbenzenesulfanate 0.80 weight section. The result was shown in Table 10.

lecyt-trimethylammonium-chloride 0.33 weight section. The result was shown in Table 10.

42] ble 10]

p.//www.ipdl.inpit.go.jp/cgi-bin/tran_web_cgi_ejje?atw_u=http%3A%2F%2Fwww4.jpd8.i... 2009/06/15

JP.11-343152,A [DETAILED DESCRIPTION]

表示 (大元	838 838 83	7 H H H H H H H A A A A A A A A A A A A	**************************************		R	2.0 de 2.
4 8 8 8 9, 8	6. 3 B	ž.	2000	後い	# 6	デデンタスンスンカンボン数ナドカウス デザンカトリメ チルアン・カンター
			3E }	A 1		
	æ:		i	ſ	,	۵
39.3	100	47.4	₹ % ~	37.76		2.0
		タチズ	マナス	43.55		r-
40		4.6×	144	4363		ಮ
1	:05	Ħ	2	4554		57
3 0 2	-	æ	×	4636		:
17, 1	375	×	æ	11.53	4.	83 C
(Θ.	::::::::::::::::::::::::::::::::::::::	×	ガシル	8	::
	24	Ħ	m	(N.47)		10 C-
			æ	23.5.6	\$	27 '4'
- 0. 23	55-1	×	æ	204%	6	6.3
(%) (%)	,,,,,,	æ	æ	, as	<u> </u>	添加器数.
太祕海2.4時間	*****	S	3			

Example 32 was rinsed and dried further, and the polyviny! butyral resin which reduced the content of sodium salt was obtained. It was 20 ppm when the sodium content of the obtained polyvinyl butyral [0243]The polyvinyl hutyral resin compounded in example 42 (preparation of polyvinyl butyral resin) resin was measured by ICP emission spectrometry. The particle diameter of sodium salt was 3.5

0244}(Production of a resin layer) Polyvinyt-butyral-resin 100 obtained weight section, On the same layer, the moisture-proof white-blush-mark sex test was done like Example 1. The result was shown weight section, dadscylbenzenesulfanic acid 0.33 weight section, and the decyl amine 0.17 weight section was carried out, and the 0.8-mm-thick resin layer was obtained. About the obtained resin canditions as Example 32, it kneaded, press forming of triethylene-glycol-di-2-ethyl-budyrate 40

dodecylhenzenesulfanic-acid 0.33 weight section and decyl amine 0.17 weight section except having used dodecylbenzenesulfonic acid 0.17 weight section and decyl amine 0.09 weight section. The [0245] The resin layer was obtained and evaluated like Example 42 instead of example 43 result was shown in Table 11.

dodecylherzenesulforic-acid 0.33 weight section and decyl amine 0.17 weight section except having used dodecylbenzenesulfanic soid 0.03 weight section and decyl amine 0.02 weight section. The 0246] The resin layer was obtained and evaluated like Example 42 instead of example 44 result was shown in Table 11.

evaluated like Example 42 except having used N.N-dimethyl ootylamine 0.17 weight section. The 10247] Instead of example 45 decyl amine 0.17 weight section, the resin layer was obtained and result was shown in Table 11.

[0248]Instead of example 48 dodecylhenzenesulfanic-acid 0.33 weight section and decyl amine 0.17 dodecylbenzenesulfanic acid 0.17 weight section and N.M-dimethyl octylemine 0.09 weight section. weight section, The resin layer was obtained and evaluated like Example 42 except having used The result was shown in Table 11.

[0249]Instead of example 47 dodsoylbenzanasulfonic-acid 0.33 weight, section and decyl amine 0.17 dedecylbenzanssuffenic scid 0.03 weight section and N.N-dimettyl octylamine 0.02 weight section. weight section. The ream layer was obtained and evaluated like Example 42 except having used he result was shown in Table 11.

dodecylbenzenesulforic acid 0.30 weight section and N.N-dimetryldodecyl amine 0.20 weight section. [0250]Instead of exemple 48 dodecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section. The resin layer was obtained and evaluated like Example 42 except having used The result was shown in Table 11.

51]Instead of example 49 dodeoy/benzenesulfonic-acid 0.33 weight section and docy/ amine 0.17 ght section. The resin layer was obtained and evaluated like Example 42 except having used lecylbenzenesulfonic acid 0.12 weight section and N.N-dimethy/dodecy/ amine 0.08 weight section. 3 result was shown in Table 11.

52]The resin layer was obtained and eveluated like Example 42 except not having used nearstive example 17 dodecylberizeresialfonic-zoid 0.33 weight section and decyl amine 0.17

nparative example 17 dodecylberizenesulfonic-acid 0.33 weight section and decy ght section. The result was shown in Table 11.

53]Comparative example 18 dodacybenzaneaulfonic-acid 0.33 weight section was not used, but reain layer was obtained and evaluated like Example 42 instead of decyl amine 0.17 weight ation except having used decyl amine 0.36 weight section. The result was shown in Table 11. 54]The resin layer was obtained and evaluated like Example 42 instead of comparative example 19 lecylbenzenesulfonic-acid 0.33 weight section and decyl amine 0.17 weight section except having addecylbenzenesulfonic acid 0.30 weight section. The result was shown in Table 11.

of dodecylbenzenesulfonic acid U.30 weight section. The result was shown in Table 13.

55]The resin layer was obtained and evaluated like Example 42 instead of comperative example 20 lecylbenzenesulfonio-acid 0.33 weight section and desyl amine 0.17 weight section except having as ordium dodecylbenzenesulfonate 0.50 weight section. The result was shown in Table 11.

56]The resin layer was obtained and evaluated like Example 42 instead of comperative example 21.

30) the resin layer was obtained and eveluated like Example 42 instead of comperative example 2 tecylbenzenesulfonlo-acid 0.33 weight section and decyl amine 0.17 weight section except having did dodecyl-trimethylammonium-chloride 0.30 weight section. The result was shown in Table 11.

bie 11]

64 20 3, 3, 5 . . 18, 5 65 65 85 8.35 .4. 3 3 3 3 数数的光光 8.8, 4 添加當數 රා ධ ස \$. \$. 39 75 ° 88 8.50 % 7.78 7.88 ස ස ස 8 0 ° 0 8, 17 0 0 6, 68 8,88 Ö Ó 1. 17 77% XFR XFR メキガーメルツ 174 X41 W.Ex 8 w 130 ¥ アデンドトロステジアンをコウムシロセイド ドゲシルベンセンスタホン級チャリロス XYX * a X * 所がCも2362数 0, 33 デッル 9. 03 FIL 13.58 8563 FF34CM2382番 9. 17 デンル ガンラ å 4. 3.3 \$ \$ \$ \$ \$ 3 3 5 9. 1. 2 9.0 添加彩数 スラサン版 アデンタインゼンスルキン製造 行いないだいまか数 FAMINISTROM 行为外的政治的数 1750人がスポンスを入機 行がかいいれば激 なったくかっていると数 io V ~~ ~ .60 2.0 30 **

58]Polyvinyl-butyral-resin 100 weight section compounded in example 50 Example 32, On the ne conditions as Example 32, it kneaded, prass forming of triethylene-glycol-di-2-ethyl-butyrate weight section, outanoic asid 0.30 weight section, and the decyl amine 0.35 weight section was ried out, and the 0.3-mm-thick resin layer was obtained. About the obtained resin layer, the isture-proof white-blush-mark sex test was done like Example 1. The result was shown in Table

59]Instead of example 51 decyl amine 0.35 weight section, the resin layer was obtained and shated like Example 50 except having used dodecyl amine 0.40 weight section. The result was wn in Table 12.

60]Instead of example 52 decyl amine 0.35 weight section, the resin layer was obtained and Anated like Example 50 except having used tetradesylamine 0.45 weight section. The result was 61]The resin fayer was obtained and evaluated like Example 50 instead of example 53 octanolo-4 0:30 weight section and decyl amine 0.35 weight section except heving used myristic acid 0.50

wn in Table 12.

x//www4.indhinnit.go.jp/cgf~bin/tran.web_cgi_ejie?atw.u=http%3A%2F%2Fwww4.jpd£i... 2009/06/15

JP.11-343152,A [DETAILED DESCRIPTION]

weight section and dodecy! amine 0.40 weight section. The result was shown in Table 12. [0252]Instead of example 54 decyl amine 0.35 weight section, the resin isyer was obtained and evaluated like Example 50 except having used N.M-dimethyldodecyl amine 0.45 weight section. The result was shown in Table 12.

[0263]The resin layer was obtained and evaluated like Example 50 instead of example 55 octanoicated 0.30 weight section and decyl amine 0.35 weight section except having used benzoic acid 0.30 weight section and dodecyl amine 0.40 weight section. The result was shown in Table 12. [0264]The rasin layer was obtained and evaluated like Example 50 except not having used comparative example 22 decyl amine 0.35 weight section. The result was shown in Table 12. [0264]The Table 12.

[Table 12]

水炭液2~地隔	₹ ₹ \$		2 4, 3	23, 7	23, 8	2.9.8	2 7. 0	
	茶和彩製	0.35	9.46	8, 18	0° 40	メギル 0. 45	0, 40	c
A	æ	æ	×	æ	æ	メチグ	æ	;
A # 1	ž	æ	æ	æ	æ	# 4 4	æ	ļ
	ä	ガンや	11.7%	\$1.66.14	\$4.64 N	松结	17.78	į
***	添加碗	8, 39	8 E .	ම ව ව	0, 50	8, 39	ය හ ස	9, S 6
なるたン数	楼板	オクダン数	ガラカン数	巻ハルルゲ	ミリスチン数	オクタン酸	妄席香靡	オクタン教
		© 23		153 151	22 NO	9	35 63	54 54
		B	Ķ.	Æ:	185	<u>.</u>		対象変

(0266)Polyvinyl-butyrai-resin 160 weight section prepared in example 36 Example 42, On the same conditions as Example 32, is kneaded, press forming of triethylene-glycol-di-2-ethyl-butyrate 40 weight section, Jf (n-butyl) phosphorio acid 0.16 weight section, and the dodecyl amine 0.14 weight section, we carried out, and the 0.8-mm-thiok resin layer was obtained. About the obtained resin layer, the moisture-proof whita-blush-mark sex test was done like Example 1. After manufacturing the glass laminate like Example 1 using the obtained resin layer and neglecting it for four weeks under the atmosphere of 50 ** and 95%RH, an adhesive (pan Merul) examination which in Example 1 was carried aut, and the adversive property of the glass peripheral part which absorbed moisture was investigated. When the periphery had a portion (pan Meru 0-1) with remarkable extolation of glass compared with the center section at this time, the width of this portion was measured and it was possiblered as exfoliation distance. The result was shown in Table 13.

[0267]The resin layer was obtained and evaluated like Example 56 instead of example 57 JJ (n-butyf) phosphoric acid 0.15 weight section and dodecyl amine 0.14 weight except having used JI (n-butyl) phosphoric acid 0.17 weight section and MN-dimethyl octylamine 0.13 weight section. The result was shown in Table 13.

[0268]The resin layer was obtained and evaluated like Example 56 instead of example 58 Jf (n-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used Jf (n-ethylhexyl) phospivoric acid 0.19 weight section and dodecyl amine 0.11 weight section. The result was shown in Table 13.

(0269)The resin layer was obtained and evaluated like Exemple 56 instead of example 59 Jl (n-butyl) shosphoric soid 0.16 weight section and dodecyl amine 0.14 weight except having used Jl (n-ethylhexyl) phosphoric soid 0.20 weight section and N.M-dimethyl octylamine 0.10 weight section. The result was shown in Table 13.

[0270]The resin leyer was obtained and evaluated like Example 56 instead of axample 80 Jl (n-butyl) phosphoric acid 0.16 weight section and dodocyl amine 0.14 weight except having used Jl (n-dodecyl) phosphoric acid 0.29 weight section and dodecyl amine 0.10 weight section. The result was shown in Table 13.

[9271]The resin layer was obtained and evaluated like Example 56 instead of example 51 Jf (n-buty!) phosphoric ecid 0.16 weight section and dodecy! emine 0.14 weight except having used Jf (2-dadecy!)

http://www.q.ipdi.inpit.go.jp/ogi-bin/tran_web_ogi_gije?atw_u=http%3A%2F%2Fwwwq.ipdi... 2009/08/15

72]The resh layer was obtained and evaluated fike Example 56 instead of example 62 Jf (n-butyf) sphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used renylphosphoric acid 0.17 weight section and dodecyl amine 0.13 weight section. The result was wn in Table 13.

73]The resin layer was obtained and evaluated like Example 56 instead of comparative example 23 n°-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used no (n°-dodecyl) sodium phosphate 0.30 weight section. The result was shown in Table 13.
74]The resin layer was obtained and evaluated like Example 56 instead of comparative example 24 n°-butyl) phosphoric acid 0.16 weight section and dodecyl amine 0.14 weight except having used kecyl-trimethylammonium-chloride 0.33 weight section. The result was shown in Table 13.

75] 5le 13]

سبدع			,	,	·	 -	·	,		
2888A	(10.33)	٥	0	0	0	٥	a	0	æ	
太後後3.4 時間	× 33	1 6. 5	13, 3	3 2, 6	14, 8	13, 4	35. 6	18.7	5.7. 6	54, 3
	彩加密数	8, 3.4	8, 13	3 8	9. 3.0	3, 8.0	9.00	8, 5, 8	0,80	6, 33
アミン	â	æ	14.47	æ	**	×	* *	×		
*		×	7 2 2 2 4 5 5 7 7 7 7 8 8 4 7 7 7 7 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9	1748						
	â	*£	***	松紅	\$34.8	9499	有行作	938	130	1649
	香龙路袋	9 T &	3, 17	9, 18	0,28	9. 8.	8. 25.	5.0	ロードゲシルリン教ナトリウム	かくいも
さ 数 数	×.	38 n-734 n-740 n, 16	カーブチル カーブチル り、17	2-21m4ve	2-1944494 2-1944594	80 m 899 m 8- 7-40 m 0.	3 8- KFOW B- KFOW 8, 23	3xx4 3xx4 6.17 898	S - 1 - 10	ドデシルトリメチルアンモニせムクロライド
	* **	カーブテル		2-154W138		かとゲアル	カンディース			**
		65 in	;~ .ua	60 60	er us	9	~~ ~	8 2	00 30	2.4

perature was held, and the reactent was deposited. Then, hold the system of reaction at 45 ** for shoi 275g of 98.9 moi to the preparation pure water 2890g of example 63(1) polyvinyl-acetal resin interlayer for glass laminates. As a plasticizer, 40 copies of triathylene glycol di-2-ethyl butyrate warming — it dissolved. Temperature control of the system of reaction was carried out to 12 **. yral resin was obtained. As for the amount of residual scotyl groups, the content [of the average iH), 8.971 copy (2,8x10,"4 mol) of magnesium 2-ethylbutanoste (carbon number 6), the ultraviolet ae "lab PURASUTO mill" Oriental energy machine company) was equipped with the T die, and on Rejadding the average degree of polymerization 1700, and the saponification degrees of polyviny re superflucus water performed ringing and desiocation for 2 hours, and white powdered polyvinyl 77%2) As opposed to 100 copies of polyvinyl butyral resin obtained by the manufacture above of idual vinyl sloohol component amount was [1700 and dogree of butyrai-izing] 2 micrometers in ticle diemeter of 20 ppm and neutralizing saft in sodium concentration 34-mol% 1-mol% 65-mol%. hed. The sodium hydroxide solution which is a neutralizer neutralized the chloride catalyst, still ditions, the mixture obtained above was extruded, the film was produced, and the interlayer for of the weight of chloride catelysts [201 g of] and the n-butylaidehyde 148g were added, this tree of polymenization of the obtained polyvinyl butyral resin] of neutralizing saft (NaCl) of the ive-mentioned 3GH was 100 ppm. Subsequently, the small extrusion machine (made by a trade ours, make a reaction complete, superfluous water washes, and unreacted n-butylaldehyde is boxylic acid, and stirring mixing was cerried out uniformly. The content of the organic acid in : the extrusion temperature of 80–180 $st\!st$ and a metallic mold outlet temperature of 200 ststabsorbent, and the antioxidant were added as metal salt (adhesive strength regulator) of as lanimates about 0.8 mm thick was obtained.

78](3) After controlling the humidity of the interlayer for glass laminates obtained by the nufacture above of the glass laminate so that water content may be 0.4 to 0.5 % of the weight in air—conditioned room, it put between two float glassea (2.4 mm in thickness), and preliminary lesion was carried out by the rolling method. Subsequently, actual adhesion of the layered product which preliminary adhesion was carried out was carried out on condition of the temperature of 130

3//www4.fodlinpit.go.jp/ogi-bin/tran_web_ogi_ejiefatw_p=http%3A%2F%2Fwww4.jpdli... 2009/06/15

JP,11-343152,A [DETAILED DESORIPTION]

** and pressure 13 kg/om² in autoclave, and the glass laminate was obtained. [0279](4) The following methods estimated the pan Meru velue of the performance of the glass laminate obtained by the evaluation above. The method of the statement estimated moisture resistance in the Example 1. The result was as being shown in Table 14.

Fig. 1856] Valuation method (1) The head struck the glass laminate which was neglected for 16 hours and carried out temperature control under pen Meru value—18**0.6 ** temperature with the harmer which is 0.46 kg, and it ground until the particle diameter of glass was set to 6 mm or feas. subsequently — following the judging standard which judges with the boundary sample which carried out grade attachment of the degree of exposure of the interlayer after glass carries out partial southing the principle of exposure of the interlayer after glass carries out partial southoun, a pan Meru value — the primary stage and (**) 50** — one month after [-] and (**) 50** — it asked about three conditions two months after — The adhasive strength of an interlayer and glass is also so large that the above—mentioned pan-mel value is large, and the adhesive strength of an interlayer and an interlayer and glass is also so small that a pan Meru value is small.

[0281]The interlayer for glass laminates and the glass laminate were obtained like Example 63 except having made metal saft of narboxylic acid as shown in Table 14 as Example 64 – a 69 adhesive—strength regulator contain.

Constant regarders for interlayer for comparative example 25 glass laminates, as metal sait of carboxylic acid, instead of 0.071 copy of magnesium 2-ethylbutanoate, the interlayer for glass laminates and the glass laminate were obtained like Example 63 except having added 0.04 copy (2.8x10 ⁻⁴ mol) of megnesium acetate (carbon number 2).

[0283]The interlayer for glass laminates and the glass laminate were obtained like Example 63 except having made metal saft of carboxylic acid as shown in Table 14 as a comparative example 26 adhesive-strength regulator contain.

[9284] The performance of the glass laminate obtained by Examples 54–69 and the comparative examples 25 and 26 was evaluated like Example 53. The result was as being shown in Table 14. Invant

Table 143

[0286]the polyvinyl butyral resin (average-degree-of-polymentation 1650 and degree % of 6.7 mol of butyral-izing.) which adjusted the combination presentation of the interlayer for example 70 glass leminates like the case of Example 65 as polyvinyl-apotal resin 32 mol of amount % and survival vinyl alcohol component amount % of residual acatyl groups of 1 mol, As the sodium content of 20 ppm, alcohol component amount % of residual acatyl groups of 1 mol, As the sodium content of 20 ppm, the third signetic glass of particle diameter of 2 micrometers of neutralizing saft, and a plasticizer, 38 copies of the thethylene glycal di-2-ethylexenoate (360). The interlayer for glass faminates was obtained like Example 63 except having considered is as 0.071 copy (2,8x10⁻⁴ mol) of magnesium 2-ethylhusanoate (carbon number 8), the ultraviolet ray absorbent and the enticxidant as metal saft of carboxylic acid. The glass faminate was obtained like Example 63 using the interlayer for glass laminate was obtained like Example 63 using the interlayer for glass laminates obtained above.

[028]]The interlayer for glass faminates and the glass faminate were obtained like Example 70 except. having made metal salt of carboxylic acid as shown in Table 15 as Example 71 and a 72 adhesive-

http://www.lipdlinpia.go.jp/ogi-bin/tran.web.cgi.ejje?atw_u=https3A%2F%2F%2Fwww4.jpdli... 2009/08/15

ength regulator contain,

:88]the polyvinyl butyrai resin (average-degree-of-polymerization 1650 and degree % of 67 mol of yrairizing) which set sodium content of neutralizing salt (sodium chlorida) to 10 ppm by washing pure water as example 73 polyvinyl-acetal resin The interlayer for glass laminates and the glass wate were obtained like Example 70 except having used 32 mol of amount % and survival vinyl ohal component emount % of residual acetyl groups of 1 mal.

89]The interlayer for glass laminates and the glass laminate were obtained like Example 70 except ring made metal salt of carboxylio acid as shown in Table 15 as Example 74 -- a 78 adhesive-

ength regulator contain.

90]in manufacture of the interlayer for comparative example 27 glass laminetes, as metal salt of inates and the glass laminate were obtained like Example 70 except having added 0.04 copy hoxylic acid, instead of 0.071 copy of magnesium 2-athyliutaneate, the interlayer for glass

1x10 -4 met) of magnesium acetate (carbon number 2).

31) The performance of the glass laminate obtained by Examples 70-78 and the comparative

imple 27 was evaluated like Example 63. The result was as being shown in Table 15,

44.00 M	カルボン製の会路路(接着力器整備)	(後数力器整備)		パンタル教	de-f	水溶器24時間
阿姆 (米加州)	※	参加盤 (ス16**キル)	S. 33	经济後人 经转换日	经转录员	* * * * * * *
363 (38)	2-258565 BERGAVAL	6, 021 (2, 8)	N°5	60	NO.	8 \$
(38) (38)	1919@x9191	0, 087 (2, 8)	S.	122	*	2 %
8G8 (8B)	2、mfをイギン数ではから 年級カリウム	9, 0 3 1 (7, 0) 9, 6 2 1 (8, 5)	163	**	ter	100 (7)
368 (88)	2-168735 Mry 198943	5, 671 (8, 8)	65	s	3	3 8
363 (38)	えってきがひゃい機でがからた-5	0, 679 (2, 8)	s ₂	et	ea.	2 18
360 (38)	2 athresis marked	3, 087 (2, 8)	15.	*	~	es es
363 (88)	Hy Meditor	3, 635 (2, 8)	150	RX	~	60 54
3 G B (E B)	ito British	9, 198 (2, 8)	w	00	~	2 6
369 (38)	2-sfackが数ではが 年数カリウム	8, 893 (2, 6) 6, 82 (2, 5)	1.7	ic:	es.	en en
3 G B (8 8)	Testerally	8, 845 (2, 8)	șe:	8 5	22	62

ythexanoate (4GO). The interlayer for glass laminates was obtained like Example 63 except having aviolet ray absorbent, and the antioxident as metal salt of carboxylic acid. The glass laminate was #3]the combination presentation of the interlayer for example 79 glass laminates --- as polyxinylsidered it as 0.079 copy (2.8x10 "4 mol) of magnesium 2-ethylpentanaste (carbon number 7), the of butyrst-izing.) 33 mol of emount % and survival vinyl alcohol component amount. % of residual tal resin --- polyvinyl butyral resin (everage-degree-of-polymerization 1720 and degree % of 66 by groups of 1 mol. As the sodium contant of 20 ppm, 100 copies of particle diameter of 2 rometers of neutralizing salt, and a plasticizer, 39 capies of tetraethylene Glico Lod'z 2ained like Example 63 using the interlayer for glass iaminates obtained above.

34]The interlayer for glass laminates and the glass laminate were obtained like Example 79 except ing made metal saft of carboxylic acid as shown in Table 16 as Example 80 – an 82 adhesivemgth regulator contain.

35]In manufacture of the interleyer for comparative example 28 glass laminates, as metal salt of boxylic scid, instead of 0.079 capy of magnesium 2-ethylpentenoste, the interlayer for glass. mates and the glass laminata were obtained like Example 79 except having added 0.04 copy

36] The performance of the glass laminate obtained by Examples 79–82 and the oxmparative mple 28 was evaluated like Example 63. The result was as being shown in Table 16. <10 4 mol) of magnesium scatate (carbon number 2).

37] ble [6]

"//www.hipdlingit.go.jp/ogi-bin/tran_web_ogi_elie?atw_u=http%3A%ZF%2F%2Fwww4.ipdil... 2009/06/15

	4.00 m	カルボン酸の金属版(後名力器整集)	3 (液体力凝散率)		パンメル数	200	女女孩 第24 為第
	条数(添加量)	## 80	\$\$ \$\$ \$\$ (×10 ' ** 4) \$\$\$ \$\$\$ \$\$\$	32 XX	经时後 人	28 W 11	88 / 5 / 5 / 5 / 5 / 5 / 5
9 / B	490 (88)	2-158415748153555	\$ 78 4 GO (88) 8-14863 30 1544 0, 0 7 8 (2, 8)	65	95	155	2 8
÷	180 (39)	2- realth Markeys 0, 0 ?	86 480 (39) 2-16007 Markey 0, 071 (2, 4)	120	*	2	9*
÷.	460 (32)	2-254045788753753	81 460 (39) 2-24944444 0. 087 (2, 8)	ws.	8	1	2.88
8 2	(88) 468 (88)	177 Betisol	6, 985 (2, 8)	,,5	S.	20	\$ 8
60 60 32 33 333	2 8 1 8 8 (3 8)	74.C\$1.200.00	0. 848 (2. 8)	.03	201	ĸ	54 124

according to pure water considering the combination presentation of the interlayer for example 83 glass laminates as polyvinyl-acetal resin 20 ppm, the polyvinyl butyral resin (average-degree-of-[9298]The content of neutralizing salt (sodium chlande) with sedium concentration by washing

31 mol \$100 copy, As a plasticizer, as metal salt of 36 copies of dihexyl horse markerel peats (DHA), ultraviolet ray absorbent and the antioxidant. The glass laminate was obtained like Example 83 using The interlayer for glass laminates was obtained like Example 63 except having considered it as the and carboxylic acid 0.071 copy (2.8x10 "4 mol) of magnesium 2-ethylbutanoate (carbon number 6), the interfayer for glass laminates obtained above.

noutralizing selt. Amount % of 1 mol of residual acetyl groups, residual vinyl alcohol component amount polymerization 1650 and degree % of 68 mot of butyrat-izing.) made into 2 micrometers of particles of

[0299] The interlayer for glass laminates and the glass laminate were obtained like Example 83 except having made metal saft of carboxylic acid as shown in Table 17 as Example 84 and an 85 adhesivestrength regulator contain,

(0300) in manufacture of the interlayer for comparative example 29 glass leminates, as metal salt of laminates and the glass laminate were obtained like Example 63 except having added 0.04 copy carboxylic acid, instead of 0.071 copy of magnesium 2-ethylhutanoats, the interlayer for glass (2.8x10 -4 mol) of magnesium acetate (carbon number 2),

[0301]The interlayer for glass laminates and the glass laminate were obtained like Example 83 except having made metal sait of carbaxylin acid as shown in Table 17 as a comparative example 30 adhesive-strength regulator contain.

(0302)The performance of the glass laminate obtained by Examples 83-65 and the comparative examples 29 and 30 was evaluated like Example 63. The result was as being shown in Table 17,

Table 17]

	% % % %	カルボン彼の会路道(接接力級整備)	(旅發力級整備)	etata art	後をメニン	**	水浴滴含土成物
	強烈 (歩加業)	蒸数	张加强(×10·4-4) 如阳 经转换人 经转换3	200	新発金人	BER B	\$6 \$7 \$7 \$7 \$7 \$7 \$7
o:	* 83 BRA (3.6)	P-1940 Methods	2-1990 1 1 1 1 1 1 1 1 1 1 1 1 2 1 3 3 1 1 1 1	15	5	*	8.8
æ	88 84 DHA (38)	くいかいをなってもとのなる	CANGESTANA 8. 063 (2. 8) S	1/2	125	*	8 %
50 50	8 8 5 BHA (3 63	2 SPACES SECTION	8-36MC503888843284 02 073 (2.8) 5	5	**	94	2.6
933 938	1 28 DHA (38)	が続づたかん	6. 046 (2. 8)	×	5	120	2.6
:= 69	CHA (SE)	797数764954	9, 958 (2, 8)	33	2	9	7 %

weight section was carried out, and the O.B-nun-thick resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1. The result was [0304]Polyviny-butyral-resin 100 weight section propared in example 86 Example 42, On the same weight section, camphor sulfortic acid 0.056 weight section, and the N.N.-dimethyl octylamine 0.044 conditions as Example 42, it kneeded, press forming of triethylane-glycol-di-2-ethyl-butyrate 40

[8308]Instead of example 87 camphor sufficies acid 0.056 weight section and N.N-dimethyl octylamine 0.044 weight section. The resin layer was obtained and evaluated like Example 56 except fraving used ivdroxyprapanesulfonic acid 6.043 weight section and N.N-dimethy! octylamine 0.057 weight section. he result was shown in Table 18. http://www4.ipdi.ingit.go.jp/egi-bin/tran.web_cgi.ejie?stw_u=https%3A%2F%2Fwww4.ipdil... 2009/08/15

107]Instead of example 89 camphor sulfonic acid 0.056 weight section and M.W-dimethyl octylemine 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used Jeoylbenzencaulfonic acid 0.06 weight section and pyridine 0.02 weight section. The result was myn in Table 18.

108]Instead of example 90 camphor sulfonic soid 0.056 weight section and N.N-dimethyl octylamine 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used tacybenzenesulfonic acid 0.061 weight section and p-toluidine 0.039 weight section. The result is shown in Table 18.

09]instead of example 91 camphor sulforic acid 0.056 weight section and N.N-dimethyl octylamine 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used -cyclohexane 2 acetio-acid 0.048 weight section and dodecyl amine 0.104 weight section. The ult was shown in Table 18.

10]Instead of example 92 camphor sulfonic acid 0.058 weight section and NN-dimethyl outylamine 44 weight section, the resin layer was obtained and evaluated like Example 86 except having used inylio acid 0.042 weight section and dodecyl amins 0.06 weight section. The result was shown in

1877. The state of comparative example 31 camphor sulfonic acid 0.056 weight section and N.M.-sottyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 96 sept having used pyridine 0.1 weight section. The result was shown in Table 18.

now having used pyronic of weight section, the result was shown in faure to.
12]Instead of comparative example 32 camphor sulforic axid 0.056 weight section and N.M.nethyl cotylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 36
rept having used salicylic acid 0.1 weight section. The result was shown in Table 18.

13]Instead of comparative example 33 camphor sulfonic acid 0.056 weight section and N.N-nethyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 86 sept having used camphor sulfone sodium 0.1 weight section. The result was shown in Table 16. 14]Instead of comparative example 34 camphor sulfonic acid 0.056 weight section and N.N-sethyl octylamine 0.044 weight section, the resin layer was obtained and evaluated like Example 86.

tept having used pyridinium chloride 0.1 weight section. The result was shown in Table 18. 15] .ble 18}

5 4. 3 2.9.8 3.5 1.9, 8 88 88 88 63 '69 '8 35. 68.8 20, 5 ري دي دي 150 150 9 8 6 3:84 然的智慧 9. 8. 2. 4 3344 9. 0.2 9. 8. 8. 8 8 8 3 ب ت ت ب . න D. 0 4 3 | R. N. 9754893483 0.0 5 6 1 N. N- 2/ FALLY FR73> 0. 0 5 0 3. 8-3/4/8/398722 イグラグム…の!! 90% S. 0 4 8 | YEVWY :>> 人に大かなみと ~ \$ \$ \$ \$ \nu_{\mathcal{L}} \nu_ 海拉部教 2 6 4 2 т, ., 23、物路スタキン数テトリウム 13,1-9904号7 二節酸 8.7 《《外科》的23/42/ 数 34 機化ビリジニウム デッタンテッスを少数 とがいながい 数 凝 参照3422数 サリチル酸 トゥッチラ数 0 30 50 ر در در æ æ 94 95 ... 70

16]Polyvinyl-butyrat-resin 100 weight section prepared in example 93 Example 42. Triethylener-201-di-2-ethyl-butyrate 40 weight section, octanale acid 0.4 weight section, On the same ditions as Example 42, it knesded, press forming of N,N-dimethyl octylamine 0.11 weight section I the 2-ethylbutanoic acid magnesium 0.037 weight section was carried out, and the 0.8-mm-thick 31//www4.ipdl.inpit_go.jp/cgi-bin/tran_web_cgi.gjie?atw_u=http%3A%2F%2Fwww4.jpdll... 2008/06/15

JP,11~343152,A [DETAILED DESORIPTION]

resin layer was obtained. About the obtained resin layer, the moisture-proof white-blush-mark sex test was done like Example 1.

[0317]The friction test was done by the following method about the glass laminate obtained by fastening the above-mentioned resin layer between the glass plates (4x4 cm) of two sheets, and obtaining a glass laminate. The obtained result was shown in Table 19.

[0318]/Friction test) After the glass faminate was immersed in underwater [with a temperature of 80 ** for one week, it dried in oven with a temperature of 80 ** for 4 hours. After repeating the process of this underwater immersion and oven drying 3 times, the existence of exfoliation of the interlayer in the circumference of a glass laminate was checked visually.

[0318] The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having changed the quantity of example 94 NN-dimethyl octylamine into 0.28 weight sections.

[0320]The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having changed the quantity of example 95 octanoic acid into 0.1 weight sections, and having changed the quantity of N.N-dimethyi actylamine into 0.08 weight sections, respectively.

(9321]Ohange the quantity of example 96 octanoic acid into 0.2 weight sections, and the quantity of NAV-dimethyl octy/amine is changed into 0.09 weight sections, respectively. The result obtained by performing production and evaluation of a resin layer and a glass laminate fike Example 93 was shown in Table 19 except having used 2-ethy/haxanoic acid magnesium 0.045 weight section instead of 2-ethylhutanoic acid magnesium 0.037 weight section.

10322)Change the quantity of example 97 octanoic acid into 0.1 weight sections, and the quantity of NA-dimethyl octylamine is changed into 0.08 weight sections, respectively. The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown in Table 19 except having used 2-ethylhexanoic acid magnesium 0.045 weight section instead of 2-ethylhutanoic acid magnesium 0.037 weight section.

1032.3]Except having changed the quentity of example 98 octancic acid into D.1 weight sections, and having used decyl amine 0.06 weight section and 2-ethythexanoic acid magnesium 0.045 weight section instead of N.N-dimethyl octylamine 0.11 weight section and 2-ethythutanoic acid magnesium 0.037 weight section. The result obtained by performing production and evaluation of a resin fayer and a glass laminate like Example 93 was shown in Table 19.

10324]Except having used di(2-ethylhexyl) phosphorio acid 0.03 weight section instead of example 99 octanoic-acid 0.4 weight section, and having changed the quentity of N.N-dimethyl octylamine into 0.02 weight sections. The result obtained by performing production and evaluation of a resin layer and a glass laminate like Example 98 was shown in Table 19.

[0328]using the glass laminate obtained by the comparative example 35 comparative example 12, the result obtained by doing only a friction test like Example 93 was looked like [Table 19], and was shown

10326]The result obtained by performing production and evaluation of a rasin layer and a glass laminate like Example 93 was shown in Tebie 19 except not having used comparative example 36 octanoic axid and N.N-dimethyl octylamine.

10327]The result obtained by performing production and evaluation of a rasin layer and a glass

laminate like Exemple 93 was shown in Table 19 except not having used comparative example 37 N.N-dimethyl octylamine. [0328]The result obtained by performing production and evaluation of a resin leyer and a glass laminate like Exemple 93 was shown in Table 19 except not having used comparative example 38

octanoio acid. {032.9}Comparative example 39 octanoio ecid and M.N-dimethyl octylamine are not used. The result obtained by performing production and evaluation of a resin leyer and a glass laminate like Example 83 was shown in Tabla 19 except having used 2-ethylhexanoio ecid magnesium 0.045 weight section

instead of 2-ethylbutanoic acid magnesium 0.037 weight section.
[0330]Instead of comparative example 40 octanoic-acid 0.4 weight section, N.M-dimethy! octylamine
0.11 weight section, and 2-ethylbutanoic acid magnesium 0.037 weight section. The result obtained by
performing production and evaluation of a resin layer and a glass laminate like Example 93 was shown
in Table 19 except having used octanoic acid 0.2 weight section and 2-ethylbexanoic acid magnesium

http://www.fipdl.ingit.go.jp/cgi-bin/tran_web_cgi.ejje?atw_u=http%3A%2F%2F%2Fwww4.jpdii... 2009/08/15

I weight section, and 2-ethylbutancic acid magnesium 0.037 weight section, The result obtained by forming production and evaluation of a resin layer and a glass laminate like Exemple 93 was shown Table 19 except having used octanoic acid 0.1 weight section and 2-ethylhexaneia acid magnesium 31]Instead of comparative example 41 octanoic-sold 0.4 weight section, N.N-dimethyl octylamine 45 weight section.

32] ible 19]

	布线数		7.87		お発致マジネシッム		於放後以人學院	
	18 45	海海岛	22.55	原约188 数	*	が対数数	× (%)	846. 2. 88
92 93	オクラン路	9. %	X. R - 394589745733	60	2-164 BR 19594	3.8.8.5	24.3	88 77
	オクタン数	, O	R, K-33548956725	2 %	2-23多 階級793953	3.537	2 2. 5	13
60 74	ガンケン袋	3 6	K.K. 35402755222	20 20 20	2-584 凝酸+19553	2.037	2 5. 3	.) .)
9	オクタン数	8 °0	8, 8-3510439-4737	, c	2-xeared Medical	5 4 6 6	1.8.8	3
3- 32	オクタン数	3 .0	K. N. 559563994733	\$ 0 8	S-MANNEY SEVENSOR S. O. A.	2.045	\$0 \$2 \$0	× ×
82	ナクタン数	9, 8	ゲンタアミン	9 0 %	3-25分445 酸795396 3. 0 4. 5	2045	3 8. 4	7
8 3	\$65.0 (2045-2040-5)6	0, 63	R, N-55-2003944783	80 .8	North Mills of their	2037	\$ 0,5) (元
00 00	ŧ	;	ł	,	1	{	33, 4	·22 54
8	}	ł	ì		2-468 1888 19455	2637	38. 45	Ε.
6- 6-5	※ハルルタ	0, 4	ş	1	2-15年 総数194895	0.627	38,6	永
8	l	ì	B. R. PISSESSATES	60	2-178 BBC14395	9.00)- 24	发
6.5	ì	1	;	ļ	KENDER WERDER	3.645	35, 2	*
*	オケケン器	8) D	1	1	R-LEMARY BRUSTAN G. 6 4	0.645	28, 7	無一二
7	おいそのか	 	i.	1	2-structor modernia	6.4.5	2 8 . 9	*

in --- warming --- it dissolved. Temperature control of the system of reaction was carried out to 12 33]adding the average degree of polymerization 1760, and the degree's of polyvinyl alcohol 275g of of chloride catalysts [201 g of] and the n-butylaldehyde 148g were added, this temperature assium hydroxide solution which is a general-purpose neutralizer neutralized the chloride catalyst, ke a reaction complete, superfluous water washes, and unreacted n-butylaldehyde is flushed. The l also white powdered polyvinyl butyral resin was obtained through rinsing and desiccation for 2 irs with superfluous water. The amount of 64-mol % and survival acetyl groups of the degrae of onification of 98.9 mol to the preparation pure water 2890g of example 100(1) polyvinyfracetal sheld, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, nation of average butyral of this resin was 1-mol %.

kness were obtained. It was 45 ppm as a result of measuring the kalium content of the obtained tecytbenzenesuffanic soid as organic acid, adding 0.25 copy of dimethyi octylamine as amine and y carrying out melt kneading with a roli mill, with the press-forming machine, press forming was erage-degree-of-polymerization 1700 and amount % of 1 mol of degree % and survival acetyl sticizer, 40 copies of triethylene glycol di-2-ethyl butyrate. After having added 0,75 cepy of ried out for 30 minutes, and 150 ** of interlayers for glass laminates of 0.76 mm of average 34j(2) As opposed to 100 copies of manufacture above-mentioned polyvinyl butyral resin ups of formation of everage butyral of 64 mol) of the interlayer for glass laminates. As a wayer by ICP tuminescence ultimate analysis.

35 it was 5 micrometers as a result of measuring the particle diameter of the potassium element ch exists in the interlayer for glass laminates obtained above using time-of-flight type secondary mass spectroscopy (TOF-SIMS).

lutes at 90 more **. Thus, this adhosion was performed for the glass laminate by which preliminary esion was carried but for 20 minutes on pressure 12 kg/cm 2 and conditions with a temperature of inate with a transparent float glass (30 cm by 30 cmx2.5 mm in thickness), After putting this in in rubber bag and carrying out, indirect desulfurization mind with the degree of vacuum of 20torr for 36(3) it inserts from both sides using the manufacture above-mentioned interlayer of a glass minutes, it moved to oven, deaerated, and the vacuum press was carried out, holding for 30

2//www4.ipdl.inpit.go_jp/cgi-bin/tran.web_cgi_ejje?atw_si=ittp%3A%2F%2F%2Fwww4.ipd£i... 2009/05/15

JP,11-343152,A [DETAILED DESCRIPTION]

38/40 ページ

135 ** in air type autoclave, and the glass laminate was obtained.

0337](4) About the glass laminate obtained by the evaluation above, the humidity test was done by the following methods. The result was as being shown in Table 20.

[0336]Based on humidity test JIS R-3212 "the safety glass test method for cars", a glass faminate is neglected for two weeks under the atmosphere of 50 **-95%RH.

then, the distance (white-blush-mark distance) of the milked portion was measured from the circumference of a glass laminate,

0.40 copy of dodecyl amine instead of 0.25 copy of dimethyl octylamine as amine. Kalium centent was 0.35 copy of dodecybenzenesulfonic acid. The interlayer for gass laminates and the glass laminate .0339]In manufacture of the interlayer for example 101 glass laminates, as organic soid, instead of were obtained like Example 100 except having added 0.30 copy of petanoic acid and having added

(0340)it was 4 micrometers when the particle diameter of the potassium element which exists in the dodecylbenzenesulfonic acid as organic acid except having added 0.20 copy of di(2-ethylhexyl) 10341] In manufacture of the interlayer for example 102 glass laminates, the interlayer for glass laminates and the glass laminate were obtained like Example 100 instead of 0.75 copy of interlayor for glass laminates obtained above was measured like Example, 100,

103423ft was 3 micrometers when the particle diameter of the potassium element which exists in the mterfayer for glass laminates obtained above was measured like Example 190 phosphoric acid. Kalium content was 55 ppm.

laminates and the glass laminate were obtained like Example 100 as an adherives regulator except 10343)In manufacture of the interlayer for example 103 glass laminates, the interlayer for glass having added 0.75 copy of potassium stearate. Kalkim content was 350 ppm.

0344]It was 8 miorometers when the particle diameter of the potassium element which exists in the interlayer for glass laminates obtained above was measured like Example 100.

10345]in manufacture of the interlayer for comparative example 42 glass laminates, the interlayer for glass laminates and the glass laminate were obtained like Example 100 except not having added the organic acid and amine of the dispersing agent. Kalium content was 47 ppm.

[0346]It was 18 micromaters when the particle diameter of the potassium element which exists in the interlayer for glass laminates obtained above was massured like Example 100

03473m manufacture of the interlayer for comparative example 43 glass laminates, the interlayer for glass laminates and the glass laminate were obtained like Example 100 not having added the organic acid and emine of the dispersing agent, and except heving changed washing time from 2 hours in 3 hours. Kallum content was 34 ppm. 0346]It was 12 micrometers when the particle dameter of the potassium element which exists in the interlayer for glass laminates obtained above was measured like Example 100.

0349]The humidity test of the glass laminate obtained by Examples 101~103 and the comparative examples 42 and 43 was done like Example 100. The result was as being shown in Table 20. 3350

Table 20]

			K.	SE 18		2	×.
		385	161	33	83	42	Ş
	アセラール高級素数	~	₩	4	*	₩.	×
-5	な格式チラール化像 (mo1光)	£.	84, 8	339	82.0 83.8 84.4	81.4	2 0
. 8	数はアセチル株数 (mo1.8)	G.	ω. 	0	6 	8 T	3,8
ä	学地度合成	982	386	1760	90%	1306	1784
25	对塞前数 (资益税)	8	40	\$	ş	ఫ	¥
	かりな人機能(1996)	35	48	93	336	47	æ
	カリラム教隆 (at)	ŝ	njg9	89	80	.	23
1	酵気性気管機の異体からの Ei化系数(mm)	1.3	250 24	65. ~	5.3	क ल	တ က်
ž	総合代定	O	٥	ं	O	×	×
						-	******

2009/06/15 http://www4.ipdl.inpit.go.jp/cgi-bin/tran_web_cgi_ejje?stw_u=http%3A%2F%2Fwww4.ipdli... JP,11-343152,A [DETAILED DESCRIPTION]

20/40 ページ

is -- warming -- it dissolved. Temperature control of the system of reaction was carried out to 12 52]adding the average degree of polymerization 1700, and the dagree's of pulyviryl alcohol 275g of 35% of chloride catalysts [201 g of] and the n-butylaidehyde 148g were added, this temperature ke a reaction complete, superfluous water weshes, and unreacted in-butylaidehyde is flushed. The flum hydroxide solution which is a general-purpose neutralizer neutralized the chloride catalyst, also white powdered polyvinyl butyral resin was obtained through rinsing and desiccation for 2 ars with superfluous water. The amount of 64-mol % and survival acetyl groups of the degree of confibration of 98.9 mol to the preparation pure water 26.90g of example 104(1) polyvinyl-acatal sheld, and the reactant was deposited. Then, hold the system of reaction at 45 ** for 3 hours, nation of everage butyral of this resin was 1-mol %.

kness were obtained. It was 30 ppm as a result of measuring the sodium content of the obtained becybenzenesultonic acid as organic acid, adding 0.13 capy of dinethyl octylamine as amine and y carrying out melt kneeding with a roll mill, with the press-farming machine, press forming was erage-dagree-of-polymerization 1700 and amount % of 1 mol of degree % and survival acetyl sticizer, 40 copies of triethylene glycol di-2-ethyl butyrate, After having added 0.75 copy of ried out for 30 minutes, and 150 ** of interlayers for glass laminates of 0.76 mm of average 531(2) As opposed to 100 copies of manufacture above-mentioned polyyinyl butyral resin ups of formation of average butyral of 64 mail of the interlayer for glass laminates. As a wayer by ICP luminescence ultimate analysis.

ch exists in the interlayer for glass laminates obtained above using time-of-flight type secondary 54lit was 4 micrometers as a result of measuring the particle dameter of the sodium element mass spectroscopy (TOF-SIMS).

utes at 90 more **. Thus, this adhesion was performed for the glass laminate by which preliminary esion was carried out for 20 minutes on pressure 12 kg/cm² and conditions with a temperature of rubber bag and carrying out, indirect desulfurization mind with the degree of vacuum of 20torr for mate with a transparent float glass (30 cm by 30 cmx2.5 mm in thickness). After putting this in in 55](3) It inserts from both sides using the manufacture above-mentioned interlayer of a glass. minutes, it moved to oven, deserated, and the vacuum press was carried out, holding for 30 ** in air type autoclave, and the glass laminate was obtained.

56](4) About the glass laminate obtained by the evaluation above, the humidity test was done by same method as Example 100. The result was as being shown in Table 21.

copy of decyl amine instead of 0.13 copy of dinethyl actylamine as emine. Sodium content was i copy of dedecythenzenesulfonic acid. The interfayer for glass laminates and the glass laminate 57]In manufacture of the interlayer for example 105 glass laminates, as organic acid, instead of e obtained like Example 194 except having added 0.30 copy of octanoic acid and having added

683t was 5 micrometers when the particle diameter of the sodium element which exists in the riayer for glass laminates obtained above was measured like Example 104.

59)in manufacture of the interlayer for example 106 glass laminates, the interlayer for glass ecylbenzenesulfonio acid as organic acid except having added 0.28 copy of di(2-ethylhexyl) mates and the glass laminate were obtained like Example 104 instead of 0.75 copy of sphoric soid. Sodium content, was 40 ppm.

603k was 2 micrometers when the particle diameter of the sodium element which exists in the rdayer for glass laminates obtained above was measured like Example 100

inates and the glass laminate were obtained like Example 104 as an adhesives regulator except 61] In manufacture of the interleyer for example 107 glass laminates, the interleyer for glass

62jit was 7 micrometers when the particle diameter of the sodium element which exists in the rlayer for glass faminates obtained above was measured like Example 104. ing added 0.50 copy of sodium stearste. Sodium content was 180 ppm.

33]In manufacture of the interlayer for comparative exemple 44 glass laminates, the interlayer for

:://www4.indl.inpit.go.jp/cgi-bin/tran_web_cgi.e.jje?atw_c+http%3A%2F%2Fwww4.indi... 2009/08/15

glass laminates and the glass laminate were obtained like Example 104 except not having added the

organic acid and emine of the dispersing agent. Sodium content was 51 ppm.

0386] in manufacture of the interlayer for comparative example 45 glass laminates, the interlayer for glass laminates and the glass faminate were obtained like Example 104 not having added the organic 0364lit was 20 micrometers when the particle diameter of the sodium element which exists in the acid and amine of the dispersing agent, and except having changed washing time from 2 hours in 3 interlayer for glass laminates obtained above was measured like Example 104

(0366)It was 13 micrometers when the particle diameter of the sodium element which exists in the interlayer for glass laminates obtained above was maasured like Exampla 104 hours. Sodiun content was 30 ppm,

0367]The humidity test of the glass faminate obtained by Examples 105–107 and the comparative examples 44 and 45 was done like Example 104. The result was as being shown in Table 21

Table 21]

			**	衰疡落		Ň	公旅館
		200	991	808	ž	3	Ş.
	アセケール基次素数	-2¢	•••	-	. 💎		=
3	平均ブチラール化度 (a) (a) (b)	9.	84. 5.	3,0	68.0	3,48	94.0
2 8	<i>後位了セチル</i> 基盤 くm c 1 %)	5.3	: :-	5.0	9 	£. 0	225
8	罗涛廉合俊	1366	1700	300	1783	1768	1230
òo	和野衛優(夏廉斯)	\$	8	ž	2	3	\$
	ナトリウム機関(pps)	8	ŝ	\$	288	ŭ,	\$
-	ナトリウム競儀(nvo))#.	64	۲-	×	13
18 00	発展性複数後の高級からの ほ状距離 (mm)	5.3	7.4	0,7	5 	eo ~rì	77
33	数金形式	0	0	0	0	×	×

examples 44 and 45 with the particle diameter of greater than 10 micrometers of the sodium element resistance so that clearly from Table 21. On the other hand, the glass laminate of the comparative 0369]The glass laminate of Examples 104-107 by this invention showed the outstanding moisture which exists in the interlayer for glass laminates had had moisture resistance.

Effect of the Invention It consists of above-mentioned composition in this invention.

hunid stracsphere, the interlayer for glass laminates and glass laminete with few white blush marks of westherability, an adhesive property, and penetration resistance, even when moreover placed into a Therefore, without spoiling basic performance required for glass laminates, such as transparency, a glass faminate edge part can be provided.

[Translation done.]

3
1/2/
C.
~~
52,A []
~~~
C V
(3)

Ą

* SECTION

111-343

ages vansed by the use of this translation. and IMPIT are not responsible for any

his document has been translated by computer. So the translation may not reflect the original icisely.

*** shows the word which can not be translated

the drawings, any words are not translated.

### **UTTEN AMENDMENT**

Written amendment?		
The same is a second to the second se	ing date May 13, Heisei 11	The state of the s

nemant to be Amended|Specification

sthod of Amendment | Change im(s) to be Amended]Claim

oposed Amendment]

plasticized polyvinyl polyvinylacetal resin films and said 0.3-0.8-mm-thick interlayer is immersed in aim 1)An interlayer for glass laminates when it is an interlayer for glass laminates which consists ** water, wherein Hayes of 24 hours after is 50% or less.

aim 2]The interlayer for glass laminates according to claim 1 whose particle diameter of sodium in an interlayer is 10 micrometers or less.

aim 3]The interlayer for glass laminetes according to claim 1 or 2 whose particle diameter of

firm saft in an interlayer is 5 micrometers or less.

aim 4]The interiayer for glass laminates according to claim 1, 2, or 3 whose sodium concentration in interlayer is 50 ppm or less.

aim SIThe interlayer for glass laminates according to claim i whose particle diameter of potassium in an interlayer is 10 micrometers or less.

aim 7] The interlayer for glass laminates according to claim 1, 5, or 6 whose potassium assium sait in an interlayer is 5 micrometers or less.

sim 6)The interlayer for glass laminates according to claim 1 or 5 whose particle Gameter of

aim 3] The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, or 7 which is a thing icentration in an interlayer is 100 ppm or less.

taining amine which is compatible in organic acid which is compatible in resin and a plasticizer. sim 9] The interlayer for glass laminates according to claim 1, 2, 3, 4, 5, 8, or 7 which is a thing staining a compound which can form sodium salt and potassium saft, and a complex. in, and a plasticizer.

ng pontaining at least one sort chosen from a group which consists of alkali metal sait and alkaline sim 10]The interdayer for glass laminates according to claim 1, 2, 3, 4, 5, 6, 7, 8, or 9 which is a th metal salt.

1 a particle diameter of 3 micrometers or less and whose alkaline earth metal salt is a thing with a aim 11)The interlayer for glass laminates according to claim 10 whose alkali metal sait is a thing ticle diameter of 3 micrometers or less.

ill motal saft of organic acid of the carbon numbers 6-16 and whose alkaline earth metal salt is the sim 12]The interlayor for glass leminates according to claim 10 or 11 whose sikali metal salt is the iline earth metal salt of organic acid of the carbon numbers 5~16.

ain 131A glass laminate characterized by making the interlayer for glass laminates according to 3, 4, 5, 6, 7, 8, 9, 10, 11, or 12 come to intervene between glass of a couple at least.

in 14]An interlayer for glass laminates whose particle diameter of sodium salt in an interlayer it is interlayer for glass laminates which consists of plasticized polyviny polyvinylanetal resin films, and

11/www.hipdlingh.go.jp/ogi-bin/tran.web.ogi.eje?atw_u=http%3A%2F%2Fwww.hipdii... 2009/06/15

JP.11-343152,A []

ベージング

Giaim 15]An interlayer for glass laminates whose particle diameter of sodium salt in an interlayer it is an interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacatal resin films, and

Olaim 18]The interlayer for glass laminates according to claim 14 or 15 whose sodism concentration in an interlayer is 50 ppm or less.

interfeyer for glass laminates which consists of plasticized polyvinyl polyvinylacetal resin films, and is Olaim 17. An interlayer for glass laminates whose sodium concentration in an interlayer it is an 50 ppm or less.

it is an interlayer for glass faminates which consists of plasticized polyviryl polyvinylacetal resin films, Olaim 18]An interiayer for glass faminates whose particle diemeter of potessium salt in an interlayer and is 5 micrometers or less.

interlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetal resin films, and is Claim 19]An interlayer for glass laminates whose potassium concentration in an interlayer it is an

nterlayer for glass laminates which consists of plasticized polyvinyl polyvinylacetal resin films, sodium particle diameter of notassium selt in an interlayer is 5 micrometers or less, and is 100 ppm or less. Ciain 201An interlayer for glass laminates whose potassium concentration in an interlayer it is an Claim 21]An interlayer for glass laminates whose potsssium concentration in an interleyer it is an interlayer for glass laminates which consists of plasticized polyviny! polyvinylecetal resin films, concentration in an interlayer is 50 ppm or less, and is 100 ppm or less.

Olaim 23] The interlayer for glass laminates according to claim 14, 15, 16, 17, 18, 19, 20, or 21 which Claim 22]The interlayer for gass laminates according to claim 14, 15, 16, 17, 18, 19, 20, or 21 which is a thing containing amine which is compatible in arganic acid which is compatible in resin and a is a thing containing a compound which can form sodium salt and potassaum salt, and a complex. plasticizer, resin, and a plasticizer.

Olaim 24]The interiayer for glass laminates according to claim 14, 15, 16, 17, 18, 19, 20, 21, 22, or 23 which is a thing containing at least one sort chosen from a group which consists of alkali metal saft and alkaline earth metal salt.

with a particle diameter of 3 micrometers or less and whose alkaline earth metal salt is a thing with a Claim 25]The interlayer for glass laminates according to claim 24 whose alkali metal saft is a thing particle diameter of 3 micrometers or less.

alkali metal salt of organic acid of the carbon numbers 5-18 and whose alkaline earth metal salt is the Claim 26] The interlayer for glass laminates eccording to claim 24 or 25 whose alkali metal saft is the alkaline earth metal selt of organic acid of the carbon numbers 5~16.

interlayer for glass laminates given in 26 come to intervene between glass of a couple at least. Claim 27]A glass laminate making claims 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, and 25 or an

[Translation dune.]

2009/06/15 http://www4.ipdi.inpit_go_go/ogi-bin/tran_web_ogi_ejie?stw_u=https33A%2F%2Fwww4.ipdi.i..